TN
1
A5
Vol. 199
NIC

TRANSACTIONS

OF THE

AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS

(INCORPORATED)

American Institute of Mining, Metallurgical, and Petroleum Engineers.

Volume 199

MINING BRANCH

1954

Mining, Minerals Beneficiation, Coal,
Industrial Minerals, Geology,
Geophysics

THIS VOLUME IS MADE UP OF PAPERS AND DISCUSSIONS PRESENTED AT MEETINGS HELD AT ST. LOUIS, FEB. 19-22, 1951; MEXICO CITY, OCT. 29-NOV. 4, 1951; CHICAGO, SEPT. 3-6, 1952; PHILADELPHIA, OCT. 30-31, 1952; LOS ANGELES, FEB. 16-19, 1953; SPOKANE, WASH., MAY 8-10, 1953; SOMERSET, KY., JUNE 12-13, 1953; HIBBING, MINN., AUG. 31-SEPT. 2, 1953; SYDNEY, NOVA SCOTIA, SEPT. 8-13, 1953; CHICAGO, OCT. 29-30, 1953; NEW YORK, FEB. 15-18, 1954; CHICAGO, Feb. 14-17, 1955.

PUBLISHED BY THE INSTITUTE
AT THE OFFICE OF THE SECRETARY
29 WEST 39TH STREET
NEW YORK 18, N. Y.

COPYRIGHT, 1955, BY THE
AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS
(INCORPORATED)

PRINTED IN THE UNITED STATES OF AMERICA

Foreword

The American Institute of Mining and Metallurgical Engineers herewith presents Transactions AIME Volume 199, Mining Branch.

This publication is a carefully edited record of noteworthy progress in the mining industry. It is hoped that the thoughtful reader will be stimulated to think of many additional opportunities for effecting improvements in technology and techniques.

Readers interested in the closely related field of extractive metallurgy will find in this volume a list of papers that will be published in Volume 200 (1954), Metals Branch.

On behalf of the Mining Branch Council, I wish to express sincere appreciation for the work of all those who made the publication of this volume possible. The quality of the contributions attests the keen intelligence and diligent effort employed by the authors and their associates in the preparation of these papers. With the accompanying discussion they add immeasurably to the sum of human knowledge.

Permit me to suggest to the reader of the succeeding pages that this book will fail to accomplish completely the mission intended unless he is thereby encouraged to undertake comparable contributions to the progress of the mineral industries.

RALPH E. KIRK, *Chairman*
Mining Branch Council

CONTENTS

MINING ENGINEERING was paged consecutively throughout 1954. Thus, in this volume of the Mining Transactions there are 11 interruptions in the pagination. The missing pages appeared in MINING ENGINEERING. This volume consists of all the Mining Transactions for 1954.

Mining

Trackless Development of an Inclined Limestone Deposit. By R. W. JENKINS 61
Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project. By ROBERT E. THURMOND, WALTER E. HEINRICHS, JR., and E. D. SPAULDING 197
Manufacture of Tungsten Carbide Tipped Drill Steel. By T. A. O'HARA 294
Mather Mine Uses Pipeline Concrete in Underground Operations. By HARRY C. SWANSON 393
Block Caving Practice at the Jeffrey Mine. By H. H. WALLER, D. L. MONROE, and D. P. R. SMYTH 510
Drilling Practice in Swedish Mining. By INGVAR JANELID 613
Ground Water Control in Underground Mining. By R. C. MAHON 632
Rock Bolting in Metal Mines of the Northwest. By LLOYD POLLISH and ROBERT N. BRECKENRIDGE 709
The NX Borehole Camera. By E. B. BURWELL, JR., and R. H. NESBITT 805
Change to Rotary Blasthole Drilling in Limestone Increases Footage, Cuts Time, Saves Manpower. By D. T. VAN ZANDT 809
Relationship of Geology to Underground Mining Methods. By GEORGE B. CLARK 812
Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes. By W. A. BOYER 989
Discussion 1101
Diamond Drilling Problems at Rhokana. By O. B. BENNETT 1186

Minerals Beneficiation

Flotation Characteristics of Pyrrhotite with Xanthates. By C. S. CHANG, STRATHMORE R. B. COOKE, and IWAO IWASAKI 209
Flotation Theory: Molecular Interactions Between Frothers and Collectors at Solid-Liquid-Air Interfaces. By J. LEJA and J. H. SCHULMAN 221
Manganese Upgrading at Three Kids Mine, Nevada. By S. J. MCCARROLL 289
Discussion 1105
A Physical Explanation of the Empirical Laws of Comminution. By D. R. WALKER and M. C. SHAW 313
Discussion 1106
Solid State Bonding in Iron Ore Pellets. By STRATHMORE R. B. COOKE and ROBERT E. BRANDT 411
Flotation of Oxidized Zinc Ores. By M. REY, G. SITIA, P. RAFFINOT, and V. FORMANEK 416
Flotation and the Gibbs Adsorption Equation. By P. L. DE BRUYN, J. TH. OVERBEEK, and R. SCHUHMANN 519
Discussion 1106
Evaluation of Sinter Testing. By E. H. KINELSKI, H. A. MORRISSEY, and R. E. POWERS 527
A Guide to the Proper Application of Classifiers. By H. W. HITZROT 534
Milling Kentucky Fluorspar Tailings. By LEMONT WEST and ROBERT R. WALDEN 542
The Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates. By F. M. LEWIS and E. C. JOHNSON 620
Methods of Charging Rods and Balls into Grinding Mills. By OSCAR JOHNSON 622
Properties of Nonmagnetic Taconites Affecting Concentration. By DONALD W. SCOTT and ADAM WESNER 635
Correlation Between Principal Parameters Affecting Mechanical Ball Wear. By R. T. HUKKI 642
Application of Closed-Circuit TV to Conveyor and Mining Operations. By G. H. WILSON 720
Tumbling Mill Capacity and Power Consumption as Related to Mill Speed. By R. T. HUKKI 728
Collection of Laboratory Dusts. By BENNY LANGSTON and FRANK M. STEPHENS, JR. 833
Rapid Estimation of Mill Product Purity by Transparency Measurement. By S. C. SUN, H. M. FISHER, and R. E. SNOW 919
Substituted Starches in Amine Flotation of Iron Ore. By C. S. CHANG 922
Adsorption of a Mercaptan on Zinc Minerals. By A. M. GAUDIN and D. L. HARRIS 925
Analysis of Variables in Rod Milling. By WILL MITCHELL, JR., C. L. SOLLENBERGER, T. G. KIRKLAND, and B. H. BERGSTROM 1001
Operation of FluoSolids Roaster at Golden Cycle. By HOWARD R. KEIL 1181
Adsorption of Dodecylammonium Acetate on Hematite and Its Flotation Effect. By A. M. GAUDIN and J. G. MORROW 1196

Coal

Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals. By SHIOU-CHUAN SUN 67
The Federal Coal Mine Safety Act. By J. J. FORBES 76
Trends in Coal Utilization and Their Effect on Coal Marketing. By CARROLL F. HARDY 299
Frontiers in Heat Extraction from the Combustion Gases of Coal. By ELMER R. KAISER 304
Discussion 1112
Effects of Oxidation of Coals on Their Flotation. By SHIOU-CHUAN SUN 396
Exploration of the Oaxaca Coal Fields in Southern Mexico. By LUIS TORON and SALVADOR CORTES-OBREGON 505
Discussion 1113
Safety in the Mechanical Mining of Coal. By W. J. SCHUSTER 524
Longwall Mining and Mechanization, with Special Reference to Nova Scotia. By FRANK DOXEY 701
Discussion 1109
Oil Spraying at the Georgetown Preparation Plant. By A. F. MEGER 724
Productivity in Mining Pitching Seams of the Canadian Rockies. By H. WILTON-CLARK 825
Underground Electrocarbonization of Coal and Related Hydrocarbons. By T. C. CHEASLEY, J. D. FORRESTER, and ERICH SARAPUU 908
Discussion 1108
Evaluating the Performance of a Cleaning Unit. By J. VISMAN 1015
Improvements in Plant and Operations at Pueblo Coal Washery. By J. D. PRICE and W. M. BERTHOLF 1190

Industrial Minerals

Trackless Development of an Inclined Limestone Deposit. By R. W. JENKINS 61
Commercial Synthesis of Star Sapphires and Star Rubies. By CLIFFORD FRONDEL 78
American Potash & Chemical Corp. Main Plant Cycle. By M. L. LEONARDI 203
New Techniques for Evaluating Natural Corundum Ores. By HOWARD F. CARL, HOWARD W. JAFFE, and ARTHUR HOCKMAN 402
Suspension Preheating of Dry Pulverized Materials. By G. K. ENGELHART 407
Leasing of Government Potash Lands. By H. I. SMITH 421
Latest Practice in Burning Cement and Lime in Europe. By O. G. LELLEP 715
Activation Energies for the Decomposition of Limestone, Dolomitic Limestone, and Dolomite. By J. H. WERNICK 730
Characteristics of Titaniferous Concentrates. By L. E. LYND, H. SIGURDSON, C. H. NORTH, and W. W. ANDERSON 817
Discussion 1116
Lithium—Northeast Brazil Is Potential Source. By W. B. MATHER 897
Phosphate Rock as an Economic Source of Fluorine. By W. L. HILL and E. D. JACOB 994
Natural Abrasives in Canada. By T. H. JANES 1010

Geology

Genetic Relations Between Granites, Porphyries, and Associated Copper Deposits. By RENO H. SALES 497
Sedimentary Rocks at Cananea, Sonora, Mexico, and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona. By ROLAND B. MULCHAY and J. RUBEN VELASCO 628
The NX Borehole Camera. By E. B. BURWELL, JR., and R. H. NESBITT 805
Origin of Uranium Deposits. A Progress Report. By D. L. EVERHART 904
Development and Construction of a Ground-Water Supply. By OWEN F. JENSEN, JR. 1093
Structure and Mineralization at Silver Bell, Ariz. By KENYON RICHARD and JAMES H. COURTRIGHT 1095
Magnetic Attraction of Stacked Drill Rods. By JOHN L. BAUM 1203

Geophysics

Copper Soil Anomalies in the Boundary District of British Columbia. By W. H. WHITE and T. M. ALLEN 49
Gravity Surveys for Residual Barite Deposits in Missouri. By P. UHLEY and LEROY SCHARON 52
Testing for Copper and Zinc in Canadian Glacial Soils. By C. T. BISCHOFF 57
The Brown Iron Ore Resources of Missouri. By EDWARD L. CLARK and GARRETT A. MUILENBURG 63
Botanical Methods of Prospecting for Uranium. By HELEN L. CANNON 217
Depth Determinations by Electrical Resistivity. By HAROLD M. MOONEY 915
Near-Surface Hydrocarbons and Petroleum Accumulation at Depth. By LEO HORVITZ 1205
Effect of a Variable Surface Layer on Apparent Resistivity Data. By HAROLD M. MOONEY 1211

Discussion of Papers Published Prior to 1954

The Use of Wooden Rock Bolts in the Day Mines. By ROLLIN FARMIN and CARVILLE E. SPARKS (1953) **196**, p. 922 1101
An Analysis of Mine Opening Failure by Means of Models. By BERNARD YORK and JOHN J. REED (1953) **196**, p. 705 1102
Cleaning Various Coals in a Drum-Type Dense-Medium Pilot Plant. By M. R. GEER, W. A. OLDS, and H. F. YANCEY (1953) **196**, p. 696 1110
Alkali Reactivity of Natural Aggregates in Western United States. By WILLIAM Y. HOLLAND and ROGER H. COOK (1953) **196**, p. 991 1114

Extractive Metallurgy

As many members of the Mining Branch are interested in the papers of the Extractive Metallurgy Division, the titles of the papers published for that Division in 1954 are listed below. These papers, and discussion, appear in Transactions (1954) **200**, Metals Branch.

Theoretical Analysis of Diffusion of Solutes During the Solidification of Alloys. By C. WAGNER.
Copper Converting Practice at American Smelting and Refining Company Plants. By F. W. ARCHIBALD. With discussion.
Mechanism and Rate-Controlling Factors in the Dissolution of Gold in Cyanide Solution. By V. KUDRYK and H. H. KELLOGG.
Reverberatory Furnace Practice at Noranda. By J. N. ANDERSON.
Desilverizing of Lead Bullion. By T. R. A. DAVEY.
Roasting Metallic Sulphides in a Fluid Column. By H. M. CYR, C. W. SILLER, and T. F. STEELE.
Equilibrium Pressure Measurements Above ZnS from 680° to 825°C. By C. LAW MCCABE.
Methods for Separating Rare-Earth Elements in Quantity as Developed at Iowa State College. By F. H. SPEDDING and J. E. POWELL.
Fundamentals of Mixing and Agitation with Applications to Extractive Metallurgy. By J. H. RUSHTON and L. H. MAHONY.
Thoughts on Lead Blast-Furnace Smelting. By L. B. HANEY and J. B. HOPKINS. With discussion.
Ionic Nature of Liquid Iron-Silicate Slags. By M. T. SIMNAD, G. DERGE, and I. GEORGE.
Developments in the Carbonate Processing of Uranium Ores. By F. A. FORWARD and J. HALPERN.

Copper Soil Anomalies in the Boundary District Of British Columbia

by W. H. White and T. M. Allen

THE Greenwood-Grand Forks area of southern central British Columbia, known as the Boundary District, has a long history of mining exploration and production. At the turn of the century this was the premier copper mining camp in the British Empire, its total production amounting to some 20 million tons. Most of this ore came from the great Granby mines at Phoenix, but the Motherlode mine at Deadwood camp, 6 miles to the west, and several mines in Summit camp, 5 miles north of Phoenix, made important contributions. The large deposits were exhausted in 1918 and the district since has seen only desultory exploration and salvage operations.

The orebodies are mineralized skarn zones in limestone members of a thick series of Upper Paleozoic sedimentary and volcanic strata. Chalcopyrite is the primary ore-mineral. Copper carbonates and silicates occur sparingly in outcrops, but the oxidized zone generally is very shallow. Much of the surface is mantled by glacial drift which in most places ranges in thickness from 2 to 15 ft. In some of the hanging valleys, however, the glacial drift may be as much as 100 ft thick and may assume drumlin-like forms.

In 1951 an ambitious program aimed at the discovery of new orebodies and important extensions of abandoned deposits was launched by Attwood Copper Mines, Ltd. In this district so thoroughly searched by an earlier generation of prospectors, any orebody which had remained undiscovered must have little or no surface indication. Consequently, in addition to the basic detailed geological work, the program of exploration included magnetometer and self-potential surveys. *Geological bets* and geophysical anomalies were tested further, prior to diamond drilling, by a study of copper distribution in tree twigs and/or in the soil. The soil sampling and analytical methods used and some of the results seem of sufficient importance to warrant this paper.

The authors had done some plant sampling in this and other districts, using the dithizone neutral-color-end-point method (Warren and Delavault, 1948, 1949; White, 1950),[1-3] but they were unfamiliar with its soil application. Finally, after much experimenting in the field, they adopted the methods described here. These methods are not entirely original or defensible on theoretical grounds, but under field conditions of rapid sampling and analysis the results are reliable enough to be of use. Fig. 1, which shows the results of duplicate analyses of duplicate soil samples taken at 50-ft intervals across an anomalous zone, indicates the relative dependability both of the sampling and analytical methods.

W. H. WHITE and T. M. ALLEN are with the Department of Geology and Geography, University of British Columbia, Vancouver, B. C.

Discussion on this paper, TP 3610L, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Feb. 16, 1953. Los Angeles Meeting, February 1953.

Sampling and Analytical Equipment

A 2-ft piece of 1-in. solid drill steel, one end sharpened to a broad, conical point. The steel is marked at 1 ft from the point.

A 2-ft piece of ½-in. black iron pipe, one end filed to a bevelled cutting edge. The pipe is marked at 1 ft 3 in. from the cutting end.

A 3-lb hammer.

A plastic or rubberized sheet about 18 in. square.

Moisture-proof assay pulp envelopes.

A 10-mesh seive made from window screen with the paint burnt off.

A small assay spatula.

A pan balance sensitive to 10 mg.

Two ignition trays about 4 in. square, made of sheet iron turned up along the edges.

A Coleman two-burner gasoline stove.

An asbestos board about 5x8 in., used as a hot plate on the gasoline stove.

A circular aluminum rack to hold 8 test tubes while refluxing (design of Almond and Morris).

Pyrex Glassware

Large refluxing test tubes, 25x200 mm, marked at 40 ml volume.

Breakers, 20 ml.

Pipettes, 1, 5, and 10-ml capacity.

Graduate, 50 ml.

Shaking cylinders, 100 ml, glass stoppers.

Burette, 25 or 50-ml capacity, with holder.

Chemical Supplies

1 N sulphuric acid.

Hydroxylamine hydrochloride, solid crystals.

Fisher Alkacid test paper.

Copper standard solution.

Dithizone standard solution 60 mg per liter.

Water reasonably free of metals.

Soil Sampling Method: The problem of how to take a soil sample is extremely crucial. The method outlined below, adopted after a number of tests, has the advantages of uniform pattern, uniform depth, and uniform size of sample.

The area to be tested was marked off by chain and compass lines 100 ft apart, normal to the strike of possible ore deposits. Numbered stakes were set at 50-ft intervals along these lines and a soil sample was taken at each stake in the following manner.

The drill steel was driven into the ground normal to the slope of the surface to the marked depth of 1 ft, moved slightly from side to side, then carefully withdrawn. The iron pipe was inserted to the bottom of this hole, tapped down to the marked depth of 1 ft 3 in. and withdrawn; the 3-in. soil plug in the

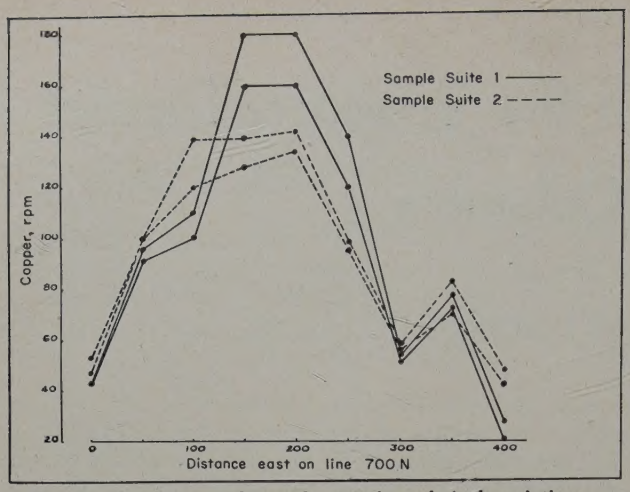


Fig. 1—Comparison of sampling and analytical variations. The dots represent analyzed samples.

end of the pipe was knocked out on the plastic sheet. Five such samples taken at each corner and at the center of a 6-ft square, combined on the sheet and transferred to a pulp envelope, made up the sample for the station. Sample weights averaged 50 g.

Preparation of Sample: At a field laboratory housed in an old shack each sample was seived and the oversize discarded. At the outset samples were pulverized, but this proved laborious and unnecessary. The screened sample was spread on a sheet iron tray, placed on the open burner of the gasoline stove, and ignited at red heat. The purpose of ignition was to break down soil aggregates and colloids and to ash organic material. The cooled sample was mixed on a plastic sheet and coned and flattened, and the 2-g sample for analysis was weighed out. This was transferred to a large test tube for digestion.

Digestion: After the addition of 30 ml of 1 N sulphuric acid, the tube containing the sample was placed in a rack on the stove and refluxed for *exactly* 30 min. The burner was so adjusted that moderate bumping agitated the contents of the tube. At the end of this period water was added to bring the volume to 40 ml, refluxing was continued for *exactly* 5 min, and the tube was set aside for half an hour to cool and settle. The procedure up to this point is described elsewhere,[4] but now a departure is made.

The next step was to transfer a 10-ml aliquot of the supernatant liquid to a 20-ml beaker to which has been added about 200 mg of hydroxylamine hydrochloride. The beaker was heated gently on an asbestos pad until the liquid had evaporated almost to dryness, that is, the center bottom of the beaker became dry. Then the beaker was removed and the contents diluted with 20 ml of water. This solution would have a pH of about 3, optimum for copper analysis using dithizone.

The procedure described in the last paragraph was found essential if all oxidizing influences were to be removed from solution, as they must be for accurate copper analyses. With this method failures due to oxidation of dithizone were less than 1 pct. On the other hand, using the method of Almond and Morris, wherein Thymol Blue indicator is added to the sulphuric solution which then is neutralized to pH.3, the authors experienced a high percentage of failures due to oxidization. Furthermore, the color of the indicator interferes with the end-point in the subsequent titration.

Titration: A suitable aliquot, usually either 1 or 5 ml, of the aqueous solution was pipetted into a shaking cylinder for titration by the neutral-color-end-point method, which is described elsewhere.[1, 2] This method is considered superior to the mixed-color methods widely used in the United States. First, partial oxidation of the dithizone, the chief danger in copper analysis, becomes immediately apparent, and second, all operators, even those slightly color-blind, get almost identical end-points.

The authors keep their supply of dithizone in solid form in folded paper packets, each containing exactly 15 mg of the reagent. Fresh solution is made up merely by dissolving the contents of a packet in a 250-ml reagent bottle of carbon tetrachloride. Each new bottle of dithizone solution is standardized, since for some unknown reason the copper equivalent may vary from one batch to the next by as much as 10 pct. To standardize, a blank to which has been added a measured quantity of standard solution of known copper content is put through the entire sequence along with the regular samples.

A three-man crew was used for the Attwood soil sampling program, two taking samples and one making the analyses. Highly-trained operators are not necessary. For example, after a short period of instruction and observation, a high-school boy became one of the best analysts. The rate averaged about 50 samples per day, a coverage at the spacings used of about 5 acres per day. Depending on rates of pay, the cost of such a program would be between $6 and $8 per acre. On a larger project costs could be reduced by greater duplication of equipment.

Examples of Copper Soil Anomalies

Soil anomalies related to known or partly-known orebodies in the Deadwood, Summit, and Phoenix camps will serve to show the kind of results which can be expected. These examples may illustrate as well some of the main characteristics of copper soil anomalies.

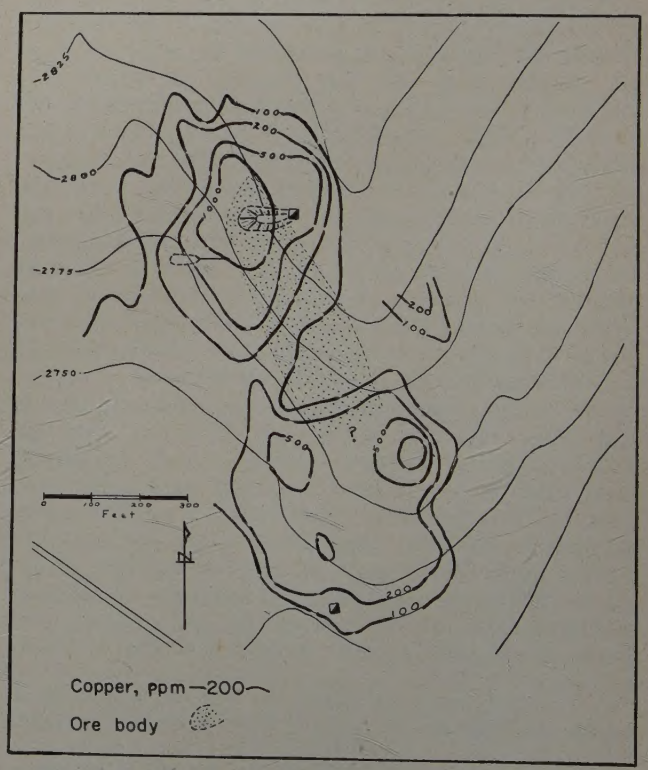


Fig. 2—Example A, Deadwood camp, showing distribution of ore and of copper soil anomaly. The heavy dashed lines are copper soil contours.

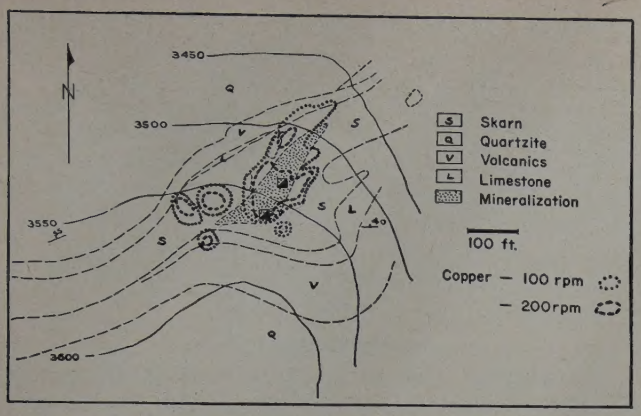


Fig.3—Example C, Summit camp, showing distribution of ore and of copper soil anomalies. Note that the copper values are much lower here than in Fig. 2.

Example A, Deadwood Camp: Fig. 2 shows the main features of this property. The low hill flanked by broad, flat-bottomed valleys is skarn containing calcite, epidote, garnet, actinolite, and hematite. Occasional flakes of malachite in outcrops and old pits indicate that the skarn in general is very slightly mineralized. Glacial drift ranges in thickness from 2 to 7 ft on the crest of the hill, becoming thicker on the flanks.

The projection of an orebody of commercial grade, as it is known at present from subsequent diamond drilling, is shown on Fig. 2. Copper mineralization, though not necessarily of ore grade, continues both North and South at least 200 ft beyond the limits shown on the map. The ore consists of the same skarn minerals found at the surface and in addition disseminated grains and small veinlets of chalcopyrite. This orebody is blind; nothing of commercial grade is known on the surface. Its closest approach to the surface is 75 ft and much of the orebody is more than 200 ft from the surface.

The distribution of copper in the soil is shown by contours on Fig. 2. Values in excess of 100 ppm are considered anomalous, and the local areas of higher copper content within the 100-ppm contour appear to have little significance. The anomaly is positioned roughly over the orebody and has a similar trend, but it has spread laterally and migrated a short distance downhill.

A magnetometer survey was made of this area. A magnetic anomaly was detected which was low and broad, roughly circular in plan and about 400 ft in diam. The orebody is more closely related in size, shape, and trend to the soil anomaly than it is to the magnetic anomaly.

The question arises as to whether the existence of this soil anomaly over an orebody which is *blind* is rational or fortuitous. It may be that the soil anomaly indicates merely the disperse copper mineralization in the skarn at the surface, which, fortunately in this instance, is related in turn to commercial ore at depth.

Example B, Deadwood Camp: A property not far from Example A will serve to show how negative results from a soil survey can be of value. The property was completely mantled by glacial drift and heavily timbered. Magnetometer work revealed several anomalies almost identical in shape and intensity to that of Example A. When the property was mapped it was found that some, but not all, of the magnetic anomalies occurred on drumlin-like hills, and doubt was expressed as to the significance of the anomalies. Consequently a soil survey was made prior to further development work. This revealed isolated high values—one sample returned over 4000 ppm copper—but no pattern of anomalous values which could be contoured rationally. A little digging proved that the magnetic charcteristics were a surface phenomenon. The magnetism was caused by magnetite sand and by large float boulders of magnetite containing some chalcopyrite, and anomalies appeared over drumlins and buried channels where the glacial drift was unusually thick.

Example C, Summit Camp: A small, relatively high-grade copper orebody in the Summit camp, which was fairly well exposed, was used to test the soil sampling and analytical methods. A lens of limestone in quartzite and greenstone has been altered in part to garnet-rich skarn, and the orebody is a well-defined mineralized zone in the skarn. The soil is rocky glacial and local drift, thin and patchy, containing much humus.

Fig. 3 shows the geology, surface outcrop of the orebody, and the copper soil anomalies. In this instance soil samples were taken at 25-ft intervals. The main anomaly is about the same size and shape as the orebody and is displaced about 75 ft downhill. The cause of the several smaller anomalies is not known.

This orebody outcrops and is of higher grade than that of Example A, yet the copper values within the anomaly are much lower; 200 ppm is the highest contour. Hence there appears to be no rational relation between copper values in the soil and those in an underlying mineralized zone.

Example D, Phoenix Camp: Fig. 4 shows an area in Phoenix camp of much interest because it adjoins a large near-surface orebody which has a fault termination. Several big gloryholes and waste dumps are only about 300 ft uphill. The bedrock in this area is completely obscured by a thick mantle of silty glacial drift, and it was hoped that a soil survey would provide some targets for the diamond

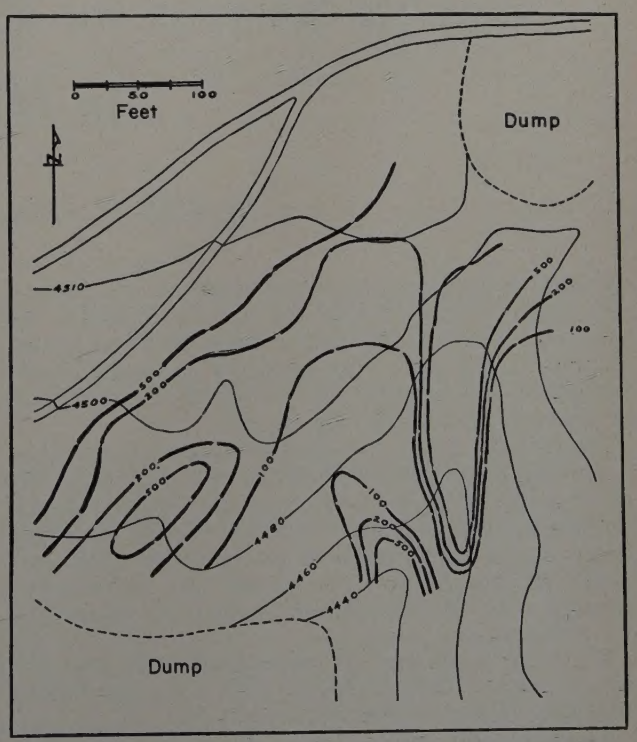


Fig. 4—Example D, Phoenix camp, showing the dispersion of copper in the soil in relation to topography. The heavy dashed lines are copper soil contours.

drill. The copper contours on Fig. 4 show that in this instance the soil survey completely failed its purpose. The copper content of the soil is anomalous throughout the area, but the distribution of copper in the soil is related solely to the topography. The greatest concentrations of copper lie along a gully which drains the large mined areas and dumps farther uphill. Evidently under such conditions of deep drift, moderate slope, and good drainage, copper in the soil can migrate differentially for distances of at least several hundred feet from its source.

Conclusions

1—Soil sampling and analysis for copper is a valid technique which properly can be included in a program of exploration for copper ore deposits.

2—Probably its greatest use will be for secondary testing of *geological bets* or anomalies obtained by geophysical methods. The cost, although not prohibitive, still is too high to permit wide use of the technique for primary prospecting.

3—Isolated spot testing is useless. Copper soil anomalies are valid only when they appear as rational contours on the map of an area that has been sampled systematically.

4—These anomalies must be interpreted with due regard to the geomorphic history of the area. They may correspond closely to the source of the copper, or, alternatively, they may have spread and migrated considerable distances. Probably in the latter instance a *tail* could be detected leading back to the source of the copper.

5—In the Boundary District of British Columbia the normal copper content of the soil is less than 100 ppm, averaging about 27 ppm. Copper values over 100 ppm can be considered anomalous. Probably in another district having different types of rocks and soils and a different climate other values would represent normal and anomalous relations.

6—A copper soil anomaly does indicate the presence of unusual amounts of copper in the underlying or contiguous bedrock, but it does not indicate, necessarily, the presence of a commercial orebody. It follows that a strong anomaly is no better indication of an orebody than a weak anomaly.

Acknowledgments

The authors wish to thank D. F. Kidd, Managing Director of Attwood Copper Mines, Ltd. for his support of this work and for permission to publish some of the results. R. H. Seraphim, Resident Manager, assisted in many ways. A. B. Young assisted with the analytical work. Material assistance and advice from Harry V. Warren and R. E. Delavault of the Department of Geology and Geography, University of British Columbia, are appreciated.

References

[1] H. V. Warren and R. E. Delavault: Biogeochemical Investigations in British Columbia. *Geophysics* (1948) **13**, No. 4, pp. 609-624.

[2] H. V. Warren and R. E. Delavault: Further Studies in Biogeochemistry. GSA *Bull.* (1949) **60**, pp. 531-559.

[3] W. H. White: Plant Anomalies Related to Some British Columbia Ore Deposits. Can. Inst. Min. Met. *Trans.* (1950) **53**, pp. 243-246.

[4] H. Almond and H. T. Morris: Geochemical Techniques as Applied in Recent Investigations in the Tintic District, Utah. *Economic Geology* (1951) **46**, No. 6, pp. 608-625.

Gravity Surveys for Residual Barite Deposits In Missouri

by Robert P. Uhley and LeRoy Scharon

TEST gravity surveys were made in the Washington County barite district of Missouri on property owned by the Baroid Sales Division of the National Lead Co. This property is located just northeast of Richwoods in the northeastern corner of Washington County, Fig. 1.

The area to which the surveys were confined comprised approximately 5000 acres, of which some 525 acres were surveyed gravitationally. Gravity observations were made at 2600 points within the area, observations being confined to relatively clear and gently sloping ground. Part of the area was heavily wooded, and the terrain too rugged for either the occurrence of a commercial barite deposit or a feasible gravity survey. The relief along the central drainage, Ditch Creek, was about 140 ft, the maximum difference in elevation being 400 ft in a distance of 3½ miles.

The purpose of the test gravity survey was to determine whether the gravity method of geophysical prospecting might be applied in the exploration for barite deposits in the Washington County barite district, since a satisfactory systematic procedure had not been devised to explore and ultimately determine barite ore reserves with desired accuracy.

No previous geophysical work with respect to barite is known to have been done in this area.

Stratigraphy: The formations over which the barite deposits occur[1] are Paleozoic in age, representing rocks involving sandstones and dolomites unconformably underlain by pre-Cambrain granites and rhyolite porphyries. The areas covered by the granite survey were directly underlain by almost

R. P. UHLEY is associated with the National Lead Co., Fredericktown, Mo., and L. SCHARON, Member AIME, is Associate Professor of Geology, Washington University, St. Louis, Mo.

Discussion on this paper, TP 3679IL, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, June 16, 1953. Los Angeles Meeting, February 1953.

flat-lying Potosi-Eminence and Gasconade dolomites which are Cambrian and Ordovician in age.

The Potosi formation is characterized by irregular masses and discontinuous beds of honeycombed drusy quartz. The weathering of the Potosi and possibly younger overlying formations has produced a residual mantle of dark red clay containing in its lower portion the residual barite which furnishes the larger production of ore.

The surface of the Potosi bedrock under the gravity areas was assumed to be somewhat flat-lying. This assumption was substantially valid but the gravity anomaly surface had a tendency to parallel the irregularities of the bedrock surface.

Ore Deposits: The residual barite occurs in a deep red plastic clay anywhere from the surface down to a depth of 20 ft. The lower portion of the deposit generally consists of red plastic clay, drusy quartz, and large blocks of barite. These blocks were sought by hand-miners who operated in the area prior to mechanization. Above this is a zone of gravel barite, disintegrated drusy quartz, and chert. Several feet of tillable leached and barren soil may occur at the surface.

The gravel barite, passed over by the hand-miners, makes the best feed for the log washers now employed to recover the barite but may be absent in some hand-mined areas.

Drusy quartz, chert, hydrous iron oxide, and a trace of galena generally are intimately associated with the ore. Some sphalerite, galena, chalcopyrite, pyrite, and barite occur along fractures in the bedrock. Origin of barite in this area is unknown.

Theoretical Considerations: The gravitational principle of prospecting is dependent on a density contrast mass of sufficient size to be measurable. In the area under consideration the basic densities involved were those of bedrock, soil, quartz, and barite. These densities were ascertained by comparison and averaging of chemical balance and weight-volume measurements with the densities of the materials in question as listed in the literature.[2] The densities were as follows: dolomite 2.88, soil 1.7, quartz and chert 2.6, and barite 4.5. The density

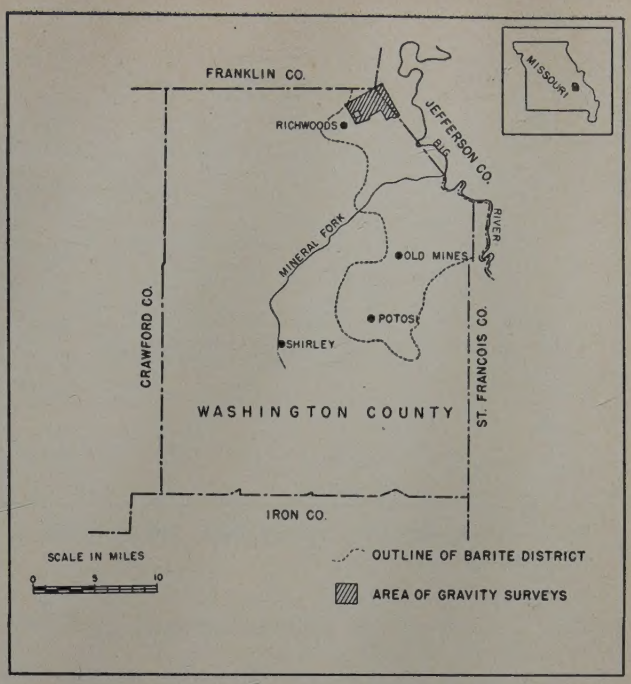


Fig. 1—Index map showing location of gravity surveys, Washington County, Mo.

contrasts with the soil were barite 2.7, quartz 0.9, and dolomite 1.2. Although barite had the highest density contrast, concentrations of quartz or dolomite in the residuum could produce a positive gravity anomaly. However, bedrock was assumed to be relatively flat-lying and quartz and chert were assumed to be uniformly distributed in the soil. These assumptions were invalid where *flint-bars* and bedrock pinnacles occurred.

Fig. 2 is a theoretical gravity anomaly[2] over a hypothetical orebody, rectangular in plan and of infinite length, having a density contrast of 0.5 and 1.0 g per cu cm. The density contrast of 0.5, which most nearly fits the case, represents a mixture of soil and barite containing about 40 pct barite by weight and produces a maximum anomaly of about 0.06 milligal. Superimposed on the theoretical

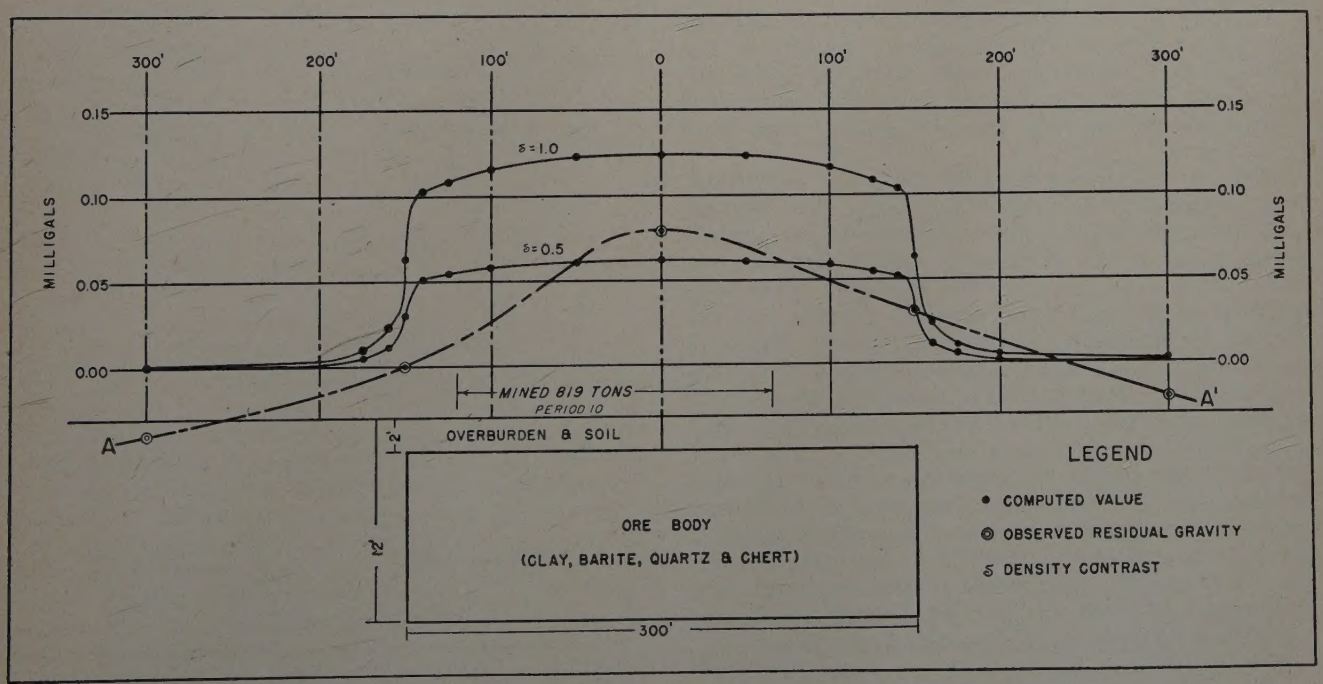


Fig. 2—Theoretical and observed gravity anomalies over a barite orebody.

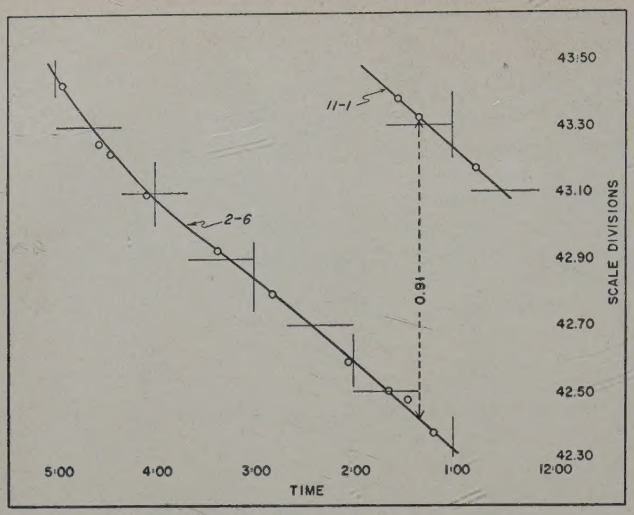


Fig. 3—Typical drift curves showing linear drift of Worden gravity meter.

profile is a measured residual gravity anomaly, A-A', over an orebody.

A series of 20 gravity observations on two stations with the Worden gravimeter[3, 4] at the Houston Technical Laboratories had a probable error of 0.02 milligal, or about 33 pct of the anticipated anomaly. Repeated readings on 19 different stations in the field had an average difference of a little less than 0.02 milligal.

Grid systems varying from 50 to 100 ft between stations were laid out with range poles and chain in small areas and with the aid of a transit in larger areas. The maximum error in station location was estimated to be 15 ft, or a maximum of 0.015 milligal. Elevations were determined to 0.1 ft or better, which was equivalent to about 0.006 milligal of Bouguer anomaly. The average density for the Bouguer correction was determined to be 2.6 by the density profile method.[5] The surveys were confined to relatively open and flat ground.

Base station observations were made at approximately hourly intervals to obtain a detailed drift correction.

Fig. 3 is a drift curve which shows the constant and linear drift of the instrument. Shocks to the gravimeter caused some erratic drift, but this was somewhat eliminated by returning the instrument to the base station after the occurrence of any kind of shock.

Results: Combined free-air Bouguer corrections were applied to the data to produce the relative gravity data. Terrain corrections were not applied in as much as the maximum terrain correction in most areas was less than the probable error of a gravity observation. The regional gravity was removed by averaging symmetrical points within a circle of 150-ft radius and subtracting from the station value. This method of removing the regional gravity was not mathematically unique but produced a reasonable residual gravity map. The relative and residual gravity data were compiled in the form of isogal maps from which an attempt was made to estimate the approximate tonnage of barite ore within the residual highs. All positive residual gravity anomalies were analyzed and anomalies of 0.03 milligal or greater, extending over four stations, were considered significant.

The only grid on which gravity measurements were made and tonnages computed and then checked by mining was the Horine tract located in the extreme southwestern corner of the survey area. On this grid 269 gravity stations were occupied.

The relative gravity map of this area, Fig. 4, shows a strong gravity gradient toward the northeast. The gravitational anomaly surface is a warped plane with a rather prominent positive nose in the central part and a negative valley in the northern part. The positive nose was partially attributed to barite mineralization.

Fig. 5 is the residual gravity map of the same area. The areas considered significant on this map were: 1—the central area centered on an 0.08 milligal anomaly, 2—the narrow positive anomalies in the southeast corner, and 3—the northwest corner. The dashed line represents the outline of the mined area for which tonnages were recorded and compared to the tonnages computed from the residual gravity anomalies. Fig. 2 showed section A-A'.

Tonnages for the mined area were computed by determination of volume between the zero gravity plane and the positive residual gravity surface.[6] The mass in 2000-lb tons was equated to 2.44 times the volume in milligal feet squared. A factor was not used for the density contrast because it was assumed equivalent to the mining recovery factor.

Table I. Estimated Tonnage vs Mined Tonnage, Horine Tract

Period	Tonnage Estimated	Tonnage Mined	Error, Tons	Error, Pct
1	620	842	— 222	— 26.4
2	860	666	194	29.1
3	270	475	— 205	— 43.2
4	20	400	— 380	— 95.0
5	860	537	323	60.2
6	840	609	231	26.4
7	310	558	— 248	— 44.4
8	280	395	— 115	— 34.4
9*	1600	531	+1069	201.0
10*	2000	819	1181	145.0
Total	7660	5832	1828	31.5

* Shallow bed rock revealed by mining.
Error in total estimated tonnage, 31.5 pct.
Error in total, excluding periods 9 and 10, 9.5 pct.

Ten mining periods were surveyed and the tonnages recorded for each period. Table I lists the tons of

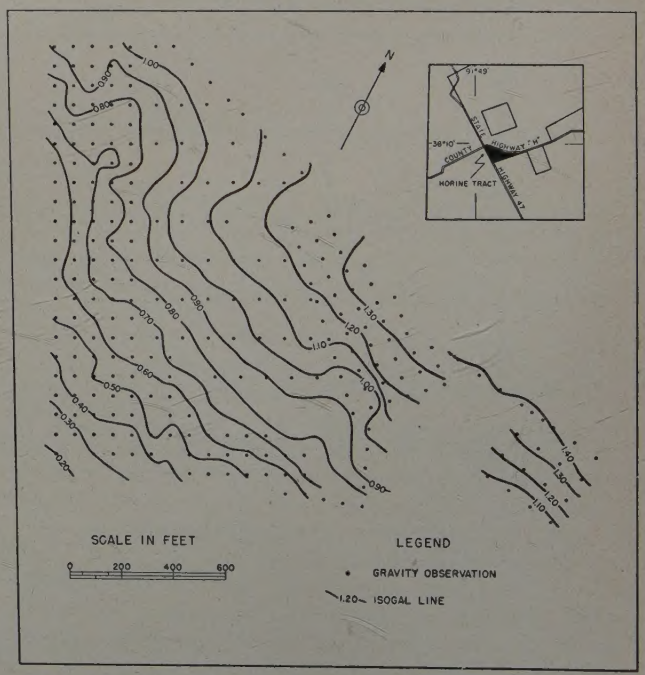


Fig. 4—Relative gravity map of Horine tract, Washington County, Mo.

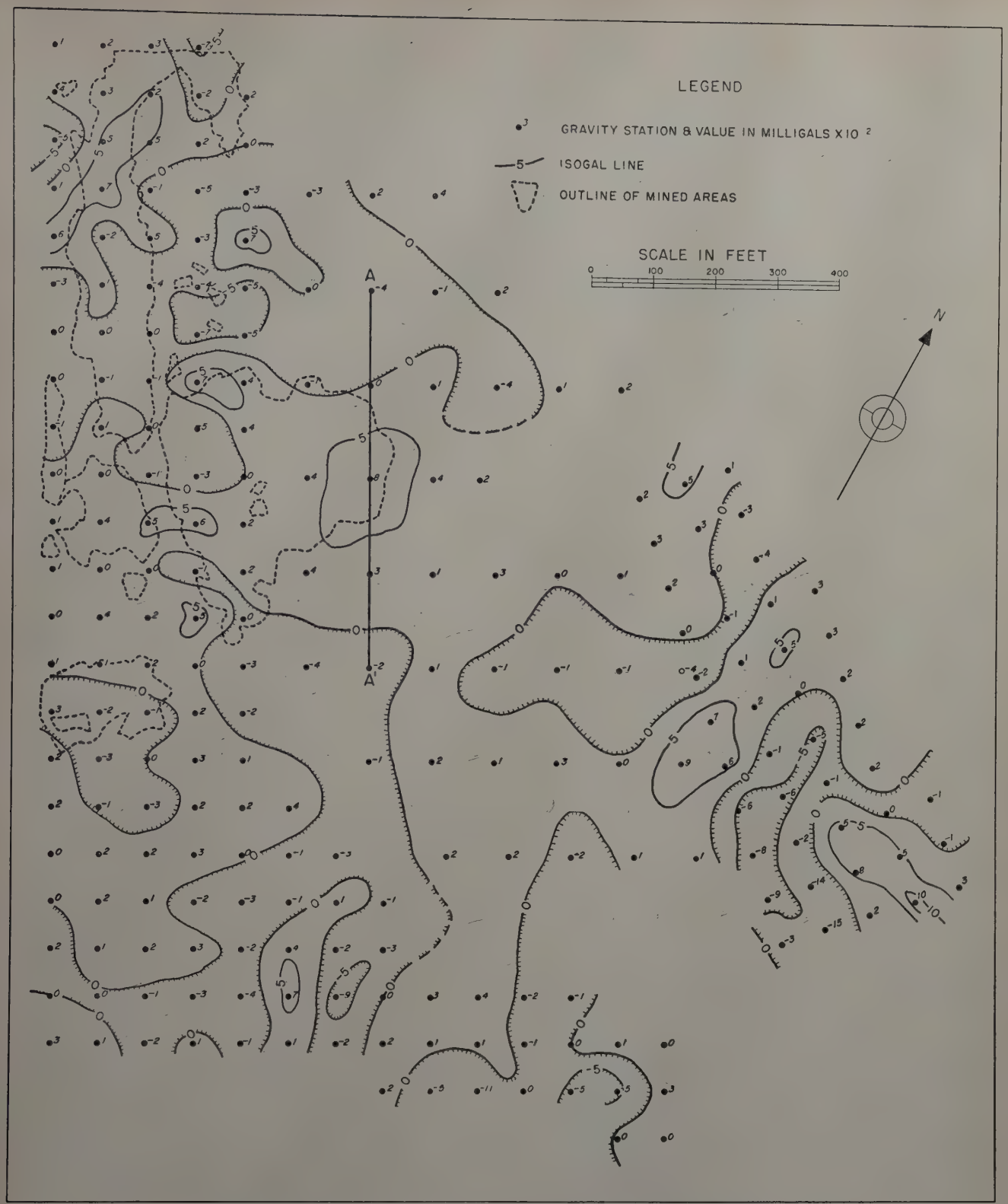


Fig. 5—Residual gravity map of Horine tract showing mined-out areas, Washington County, Mo.

barite recovered for each mining period vs the tons computed from the gravitational data. This gave an error in the total estimated tons of 31.5 pct. It should be noted that the last two periods were influenced by shallow bedrock; if they are excluded, error in total estimated tons is only 9.5 pct.

For contrast purposes typical relative and residual isogal maps prepared from one of the featureless areas are shown in Fig. 6. The one station low noticeable on the 1.9 milligal contour line was attributed to error. The small highs and lows on the residual map were considered insignificant. Test pits in the area were not recommended; however, they were made to test geophysical conclusions. The pitting did not reveal commercial mineralization but did aid in evaluation of gravity data in other areas.

Numerous test pits were dug to check gravitational results in other areas. The relative gravity maps of these areas were almost featureless, indicating the absence of large density contrast masses. Fig. 7 is a bar graph illustrating that in areas of

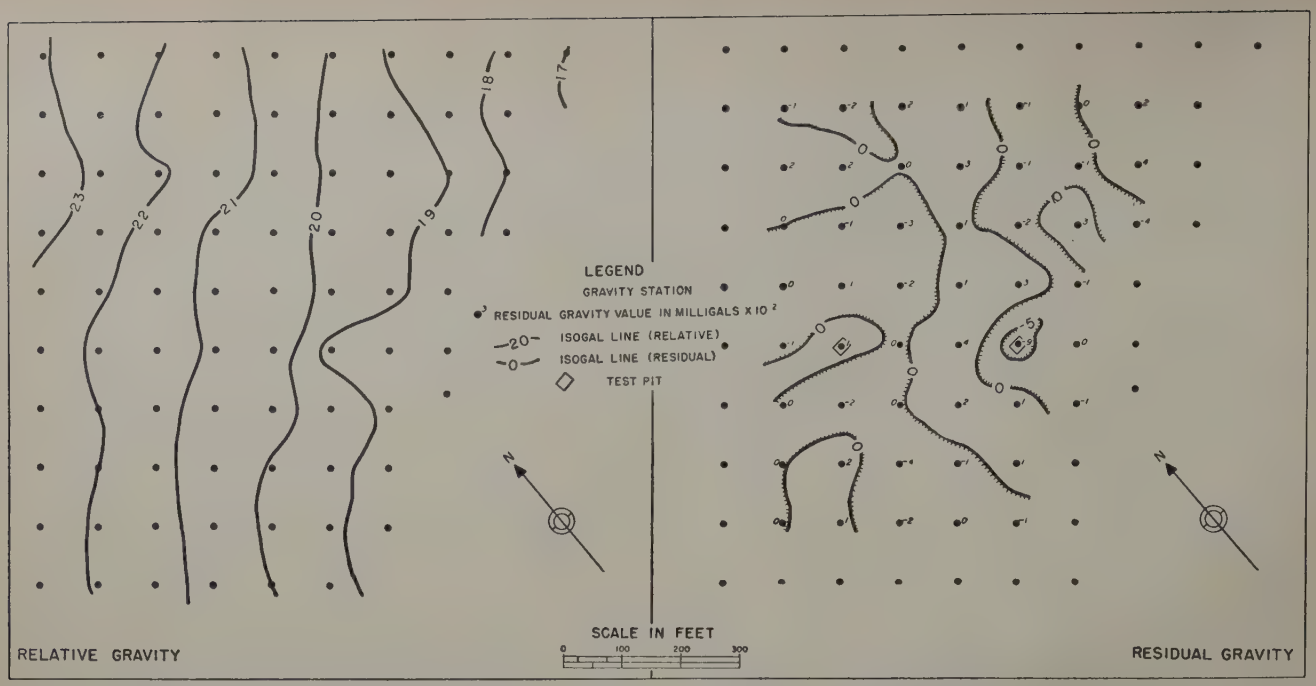


Fig. 6—Relative and residual gravity maps of a non-mineralized area, Washington County, Mo.

non-commercial deposits there was also some correlation between positive residual gravity anomalies and small areas of mineralization; however, these small pods of barite could not be located with much confidence by use of a 100-ft station interval. The graph also indicates that bedrock pinnacles were a significant factor in producing positive residual gravity anomalies.

No large concentrations of barite ore were revealed by the gravity method over the some 525 acres surveyed except that in the Horine tract. Conclusions and recommendations based on gravity observations were corroborated by test pitting. The accumulated data indicate that a large commercial body of residual barite can be outlined and the tonnages therein computed with an expected error of 35 pct or less by use of a gravity survey at proper station intervals, or by returning to increase the station in interesting areas.

The gravity data must be checked by test pitting to determine the cause of a residual gravity anomaly, because these anomalies were found to be attributable to one or more of the following factors: 1—barite mineralization, 2—bedrock pinnacles and knobs, 3—flint-bars, and 4—unknown concentrations of mass and/or probable error.

The results strongly suggest that areas lacking significant positive gravity anomalies are devoid of near-surface barite bodies of commercial size and may be eliminated from further consideration.

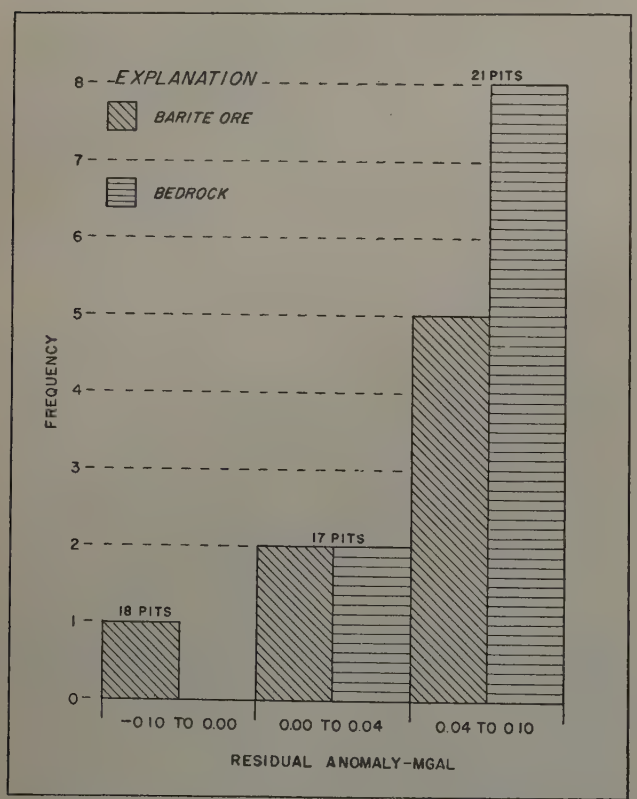


Fig. 7—Bar graph showing correlation between positive residual anomalies and small areas of barite ore, Washington County, Mo.

Acknowledgments

The authors gratefully acknowledge the permission granted by the Baroid Sales Division of the National Lead Co. to present this paper. Also they wish to express appreciation for the mining data and all other information and aid furnished by the Fountain Farm Office of the Baroid Sales Division. The writers are indebted to Rollyn P. Jacobson for the drafting of the illustrations.

References

[1] W. A. Tarr: The Barite Deposits of Missouri. *Economic Geology* (1919) **4**, pp. 46-67.

[2] C. A. Heiland: *Geophysical Exploration*, pp. 77-84, 150. Prentice-Hall Publishing Co., New York, 1940.

[3] M. B. Dobrin: *Introduction to Geophysical Prospecting*, pp. 57-58. McGraw-Hill Book Co., New York, 1952.

[4] G. P. Woollard: The Gravity Meter as a Geodetic Instrument. *Geophysics* (1950) **15**, pp. 1-30.

[5] L. L. Nettleton: *Geophysical Prospecting for Oil*, pp. 57-58, 11-154. McGraw-Hill Book Co., New York, 1940.

[6] Sigmund Hammer: Estimating Ore Masses in Gravity Prospecting, *Geophysics* (1945) **10**, pp. 50-62.

Testing for Copper and Zinc In Canadian Glacial Soils

by C. T. Bischoff

This paper describes the results of testing with colorimetric methods, using "dithizone", soil samples taken over various known copper and zinc deposits covered by glacial till. Variation in results is shown for different depths and types of overburden as well as grade and type of deposit. Limiting factors for the practical application of this work are discussed. The method has proved successful for copper and zinc deposits under clay and fine sand with depths up to at least 30 ft but fails in coarse sand and gravel unless depths are very shallow. Swamps require penetration to the underlying subsoil for sample material.

DURING the past few years geochemical testing for traces of heavy metals in water, soil, rock, and vegetation has aroused increasing interest. Various techniques for field and laboratory determinations have been described in the literature, as have some of the results of application. However, little information has been published regarding application to glacial terrain. Over 90 pct of the Canadian Shield is overburdened, resulting in high costs for mineral exploration once the outcropping ore deposits have been found. This has been largely responsible for the widespread application of mining geophysics in Canada, permitting concentration of effort on area or zones considered most favorable. However, geophysical interpretation has its limitations, since at least 90 pct of the structures indicated thereby contain no valuable minerals. A more direct approach is therefore highly desirable. If underlying base metal mineralization can be detected by sampling soil or vegetation at moderate cost this should prove of great help in locating new discoveries or extending known ones.

A large proportion of Canadian ore deposits contain appreciable copper or zinc, even where these are not the principal economic metals. Consequently the field of application is a broad one.

This paper deals with investigations along these lines with particular emphasis on soil sampling. The problems involved from the start were:

1—Which types of glacial soil, if any, would best lend themselves to testing, and conversely which types might be considered relatively hopeless?
2—Through what depths of overburden could significant mineralization be detected?
3—What sampling techniques would prove economically applicable? This included minimum depth of sample and maximum spacing that could be allowed.

C. T. BISCHOFF is a Mining Engineer, Rouyn, Quebec, Canada.
Discussion on this paper, TP 3676L, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Jan. 26, 1953. Revised Aug. 31, 1953. Los Angeles Meeting, February 1953.

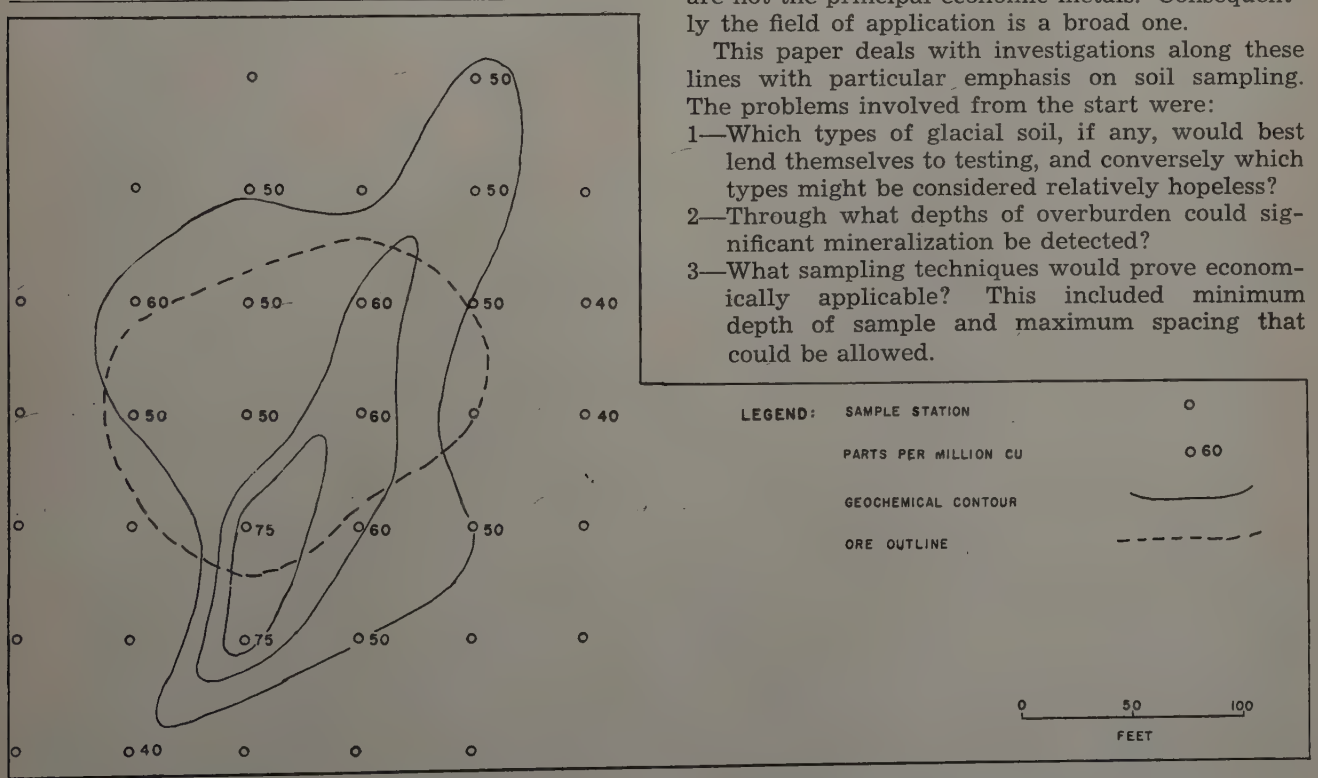


Fig. 1—Geochemical soil test. Overburden: clay, 25 to 35 ft. Mineralization: low-grade, medium-size replacement pyrite, chalcopyrite.

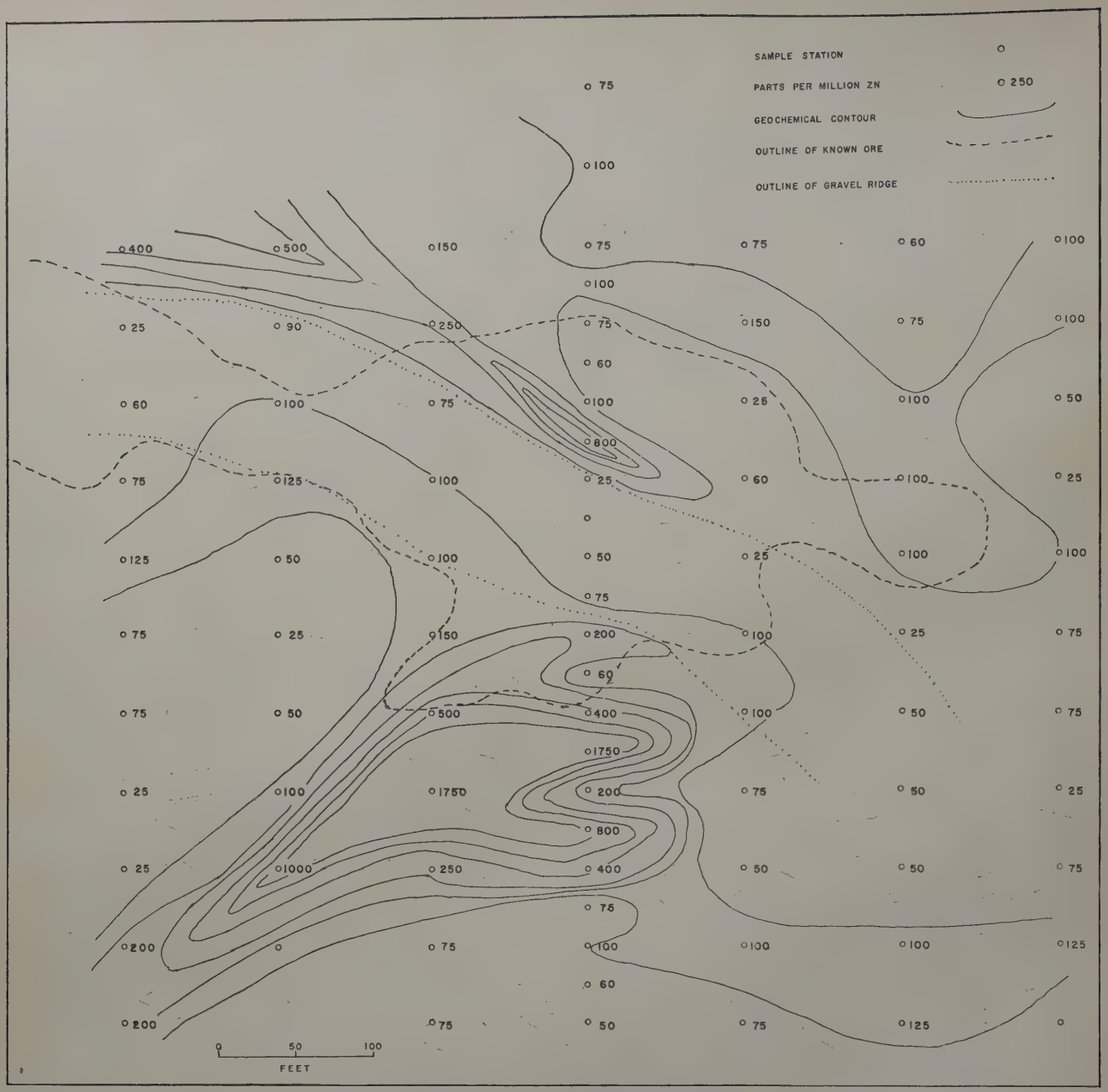


Fig. 2—Geochemical soil test. Overburden: clay, 5 to 20 ft. Mineralization: large, low-grade replacement pyrite, sphalerite. Slope: gently north and south from gravel ridge.

4—How would size of deposit and type of mineralization affect efficiency?

5—What effect might be expected from float and salting by various agents, including drainage, in the way of providing false anomalies not related to underlying or adjacent mineralization?

Soil Types

It soon became apparent that the most favorable soil types were fine-grained homogeneous clay and sand. Note in Fig. 1 the anomaly caused by low-grade copper through 30 ft of clay. Other workers have found shales tend to accumulate heavy metal in trace amounts, and there is a similar tendency in clay. Gravel and coarse sand on the contrary proved very unfavorable, probably because of rapid drainage. Note blanketing effect of sand and gravel ridges in Figs. 2 and 3. Swamps, which are common in the Canadian Shield, proved unfavorable as far as the humus and black muck went. However, it was found that if samples were taken from the underlying clay or sand, results were dependable.

The depth of favorable overburden through which ground water would bring appreciable quantities of heavy metals to surface was surprising. The practical maximum overburden is now considered to be 30 to 50 ft for clay and 20 to 30 ft for fine sand. It has also been found that there is a practical minimum depth required for efficient soil testing. Where the overburden is less than 3 to 5 ft it now appears preferable to test vegetation. More will be said of this later.

Sampling Technique

Samples of surface material have proved erratic under northern conditions and it is better to take subsoil lying below root growth or surface debris. However, in glaciated terrain the subsoil is seldom

more than 1 ft below surface except in swamps, where on one occasion it was necessary to go to a depth of 40 ft below surface.

Spacing requirements depend partly on dimensions of the source (size of deposit) partly on depth of overburden. However, a spacing of 50 ft along lines 300 ft apart is generally adequate for reconnaissance, and there was no occasion to sample closer than 25 ft.

A few ounces of material are plenty for making determinations and reserving a portion for reference. Samples are taken with a short piece of ½-in. pipe welded to the end of a 6-ft rod. This tool is inserted into a hole in the ground made with a length of bar or diamond drill rod forged to a dull point. It is then given a few blows to ensure entry into fresh undisturbed soil. One side of the pipe is ground away so that on insertion of the tool soil passes through until the tool reaches maximum depth. On withdrawal, this deepest material remains in the pipe and can be easily removed into a sample envelope.

Determinations of zinc and copper content of the soil are based on techniques described by Huff, Lakin, and others in publications of the U. S. Geological Survey and in *Economic Geology*. These techniques have been summarized in a compilation by Lakin, Almond & Ward published by the Northwest Mining Association, Spokane, Wash. Methods concerning copper and zinc have been modified considerably in the interests of convenience, speed, and accuracy. However, as the work is commercial and competitive, these modifications cannot yet be described in detail.

It must suffice to say that the color comparisons involved in the usual zinc *dithizone* determinations have been found reliable, provided both dithizone and buffer solutions are pure and fresh. However, it has been discovered that copper is more amenable to a neutral endpoint method. This can be made extremely sensitive if pH control is adequate.

Results can be made to check within 20 pct, and as anomalous values over significant mineralization have been found to be from 3 to 10 times background, such accuracy is adequate.

Parts per million of zinc and/or copper are plotted for each station on a suitable scale and contoured for interpretation.

Where anomalous metal values occur in the soil their pattern almost invariably shows some deformation or drift caused by surface or subsurface drainage. However, in shape, extent, and amplitude they have proved remarkably similar to published results of work in residual soils.

This brings up the interesting question of how much of the results obtained in residual soils may be due to underlying mineralization rather than to original metal content of the soil itself which may have been leached and dissipated. However, as work has been largely confined to transported soils, there is no answer at present.

Effect of Extent and Type of Mineralization

It appears logical that anomalous results found at surface will be proportional to the amount of metal made available through solution and transportation to its present site. Thus a large low-grade deposit will provide more effect than a small high-grade one. Shearing or other fracturing which provides fresh surface and permits access to solutions will increase the effect, and this will be multiplied if pyrite or pyrrhotite are present to provide acid for solution.

Fig. 3 shows results over somewhat lower grade material (with abundant pyrite) than is indicated in Fig. 4. Fig. 4 shows results where relatively high-grade copper (chalcopyrite) ore contains very little pyrite or pyrrhotite.

Deep overburden will disperse the values over a greater area, while a permeable layer of sand or gravel under clay may permit transportation for some distance before metal solutions reach surface. Note in Fig. 5 transportation of values away from the source.

Some difficulty has been experienced in taking satisfactory samples where the surface is covered with boulders or overburden is very shallow. In such cases biogeochemistry should prove more effective.

The experience of the writer and his co-workers with this method has been limited to date, but it appears to have few serious disadvantages. It cannot be recommended over swamps, as the roots of trees do not usually penetrate to the underlying subsoil. Open fields and recent burn fail to provide the necessary trees. However, it can still be applied to a large portion of the potential area of the Canadian Shield. It may prove more efficient than soil testing over coarse sand and gravel as the vegetation may tend to accumulate such solutions as pass through. However, this is only a conjecture so far. Certainly it is more easily applied under winter conditions, when the ground is frozen and soil sampling becomes difficult. Fig. 6 shows the results of sampling birch twigs over and adjacent to low-grade copper and zinc with abundant pyrite and pyrrhotite. Results in ppm zinc in ash are very high, as is characteristic of birch. The lobe of very high values southeast of the shaft is probably due to drainage from the dump. However, high values

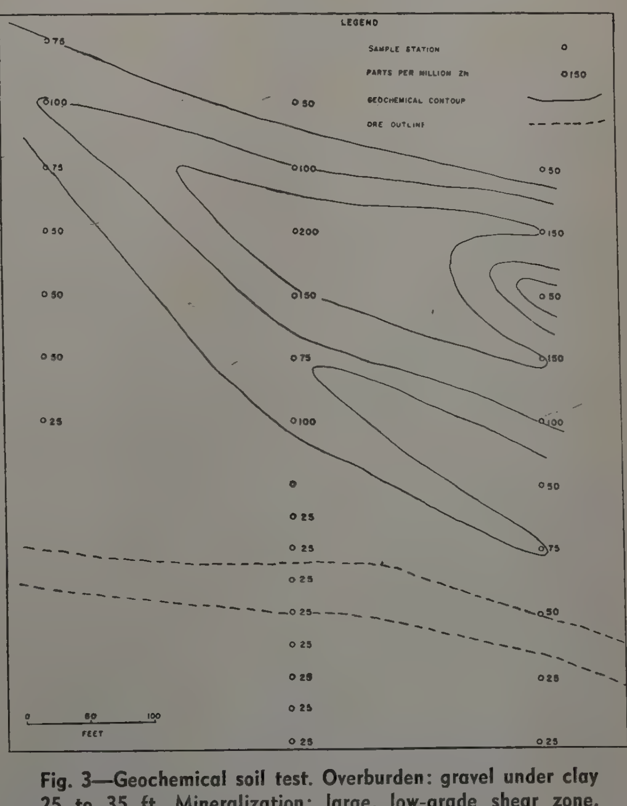


Fig. 3—Geochemical soil test. Overburden: gravel under clay 25 to 35 ft. Mineralization: large, low-grade shear zone, pyrite, sphalerite. Slope: gently north.

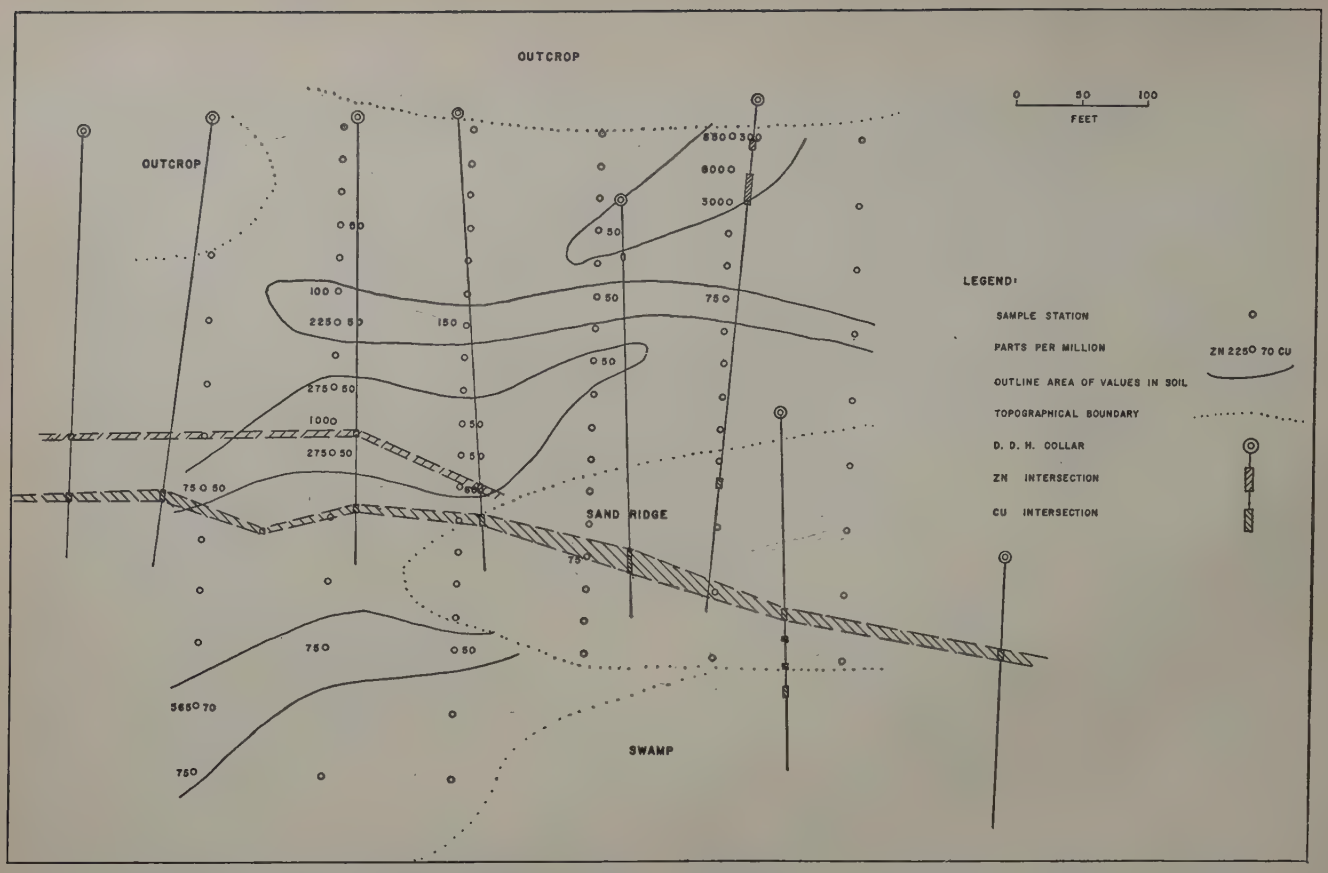


Fig. 4—Geochemical soil test. Overburden: sand, 5 to 40 ft. Mineralization: low-grade, short wide lenses, pyrite, chalcopyrite. Slope: surface, 5 pct south; bedrock, 10 pct south.

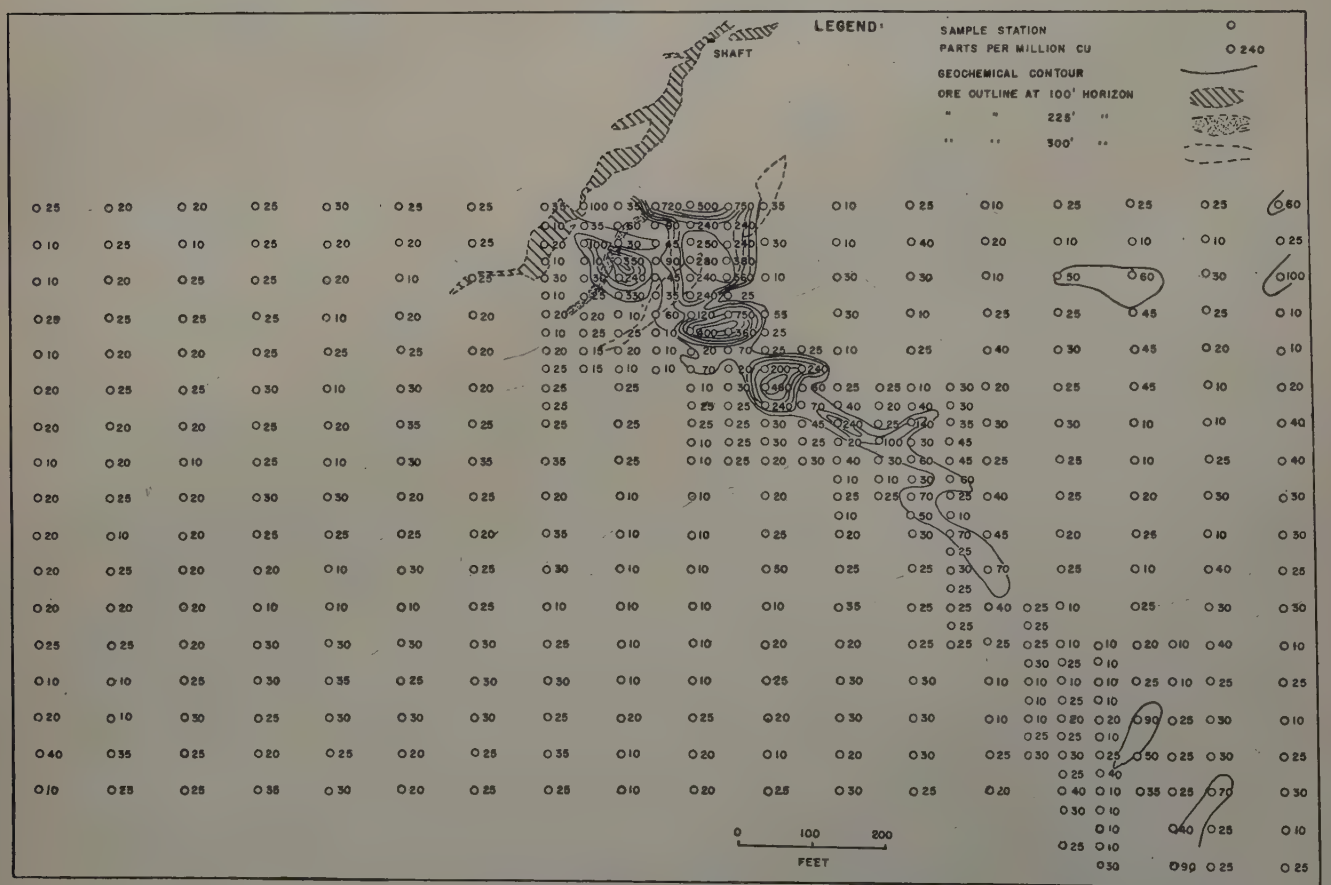


Fig. 5—Geochemical soil test. Slope: surface, 5 pct south.

in the upper left-hand corner cannot be explained by any known occurrence and may indicate additional mineralization.

While various species of trees differ in their affinity for copper or zinc, many species are sufficiently alike to be interchangeable.

Conclusions

Testing of soil and vegetation for traces of heavy metals in glaciated terrain is quite feasible under certain conditions. The overburden should be fine-grained and of moderate depth. Swamps may be sampled if the underlying subsoil can be reached. Type and size of mineralized structure as well as type and depth of overburden must be considered in assessing the applicability of the method. The ranges of anomalous values found over or near significant mineralization are usually three to ten times background, permitting colorimetric analyses to be applied.

Biogeochemistry may be substituted for soil testing except over swamps and may prove more efficient in the case of poor surface conditions or winter operations.

As is the case in geophysical work, relatively large areas should be covered by a pattern of samples, so that a picture of heavy metal distribution may be obtained. A few scattered samples can have little value and may be quite misleading.

In general geochemical techniques are more laborious on a per reading basis than most geophysical methods. Consequently they tend to be more expensive. Some very optimistic cost figures have been set forth in some publications on this subject. However, these appear to exclude indirect costs such as research, supervision, and interpretation of results, which are important items.

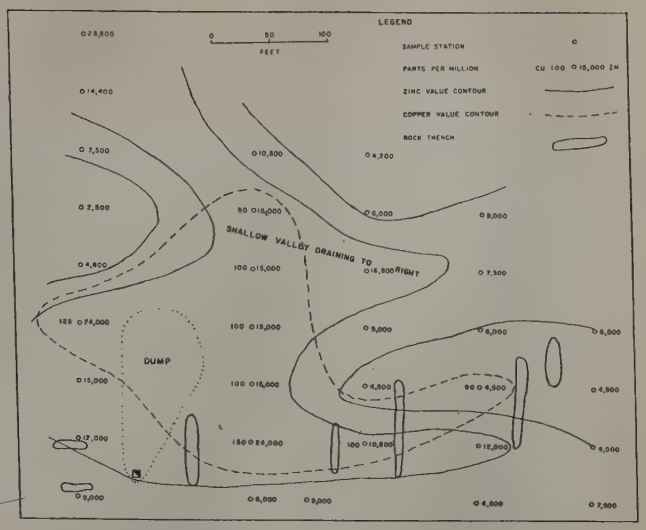


Fig. 6—Biogeochemical test. Samples: white birch, second year stems. Overburden: clay, 0 to 20 ft. Mineralization: replacement lenses are pyrite, pyrrhotite, chalcopyrite, sphalerite.

Consequently these techniques may find their best application in checking of geophysical anomalies and apparently favorable geological structures. Nevertheless, there are some conditions under which geophysical methods are quite ineffective, not to say misleading, and in such cases geochemical prospecting may provide a more efficient means of searching for buried ore deposits.

Trackless Development of an Inclined Limestone Deposit

by R. W. Jenkins

TRACKLESS equipment is being used by the Coplay Cement Manufacturing Co. to develop a folded limestone deposit economically and safely with inexperienced men. Cost and quality of development stone results in a saving when it is used instead of outside sources of limestone. This favorable cost has been attained by following a methods development and training program described in this paper. It is expected that further savings in favor of the mined stone will be made when stopes now being prepared are in production. The present equipment will be used for loading from stope draw points.

The limestone is blended with quarried *cement rock* to serve as raw material for Portland cement, first produced in this country by Coplay Cement Manufacturing Co. from a process developed by its president, David O. Saylor, in 1873. For many years the cement rock alone or with a small amount of added limestone was used for raw materials. Later as specifications were raised and the richer portions of quarries were depleted, limestone from outside sources was required in increasing amounts. In 1925 D. J. Uhle, the general manager, began to prospect company property for limestone.

It was known that the argillaceous cement rock was underlain by crystalline high calcium limestone. After an extensive drilling program it was found that there were large reserves of limestone underlying a recumbent syncline exposed in the south wall of the company's No. 3 cement rock quarry. A quarry was started on the vertical limb of this fold in 1927, and by 1930 the full requirement of the plant was met.

After 1945 production diminished because of the occurrence of dolomite beds in the south wall. The quarry floor was then 135 ft below the cement rock level and 285 ft below surface. In 1949 Gerald F.

R. W. JENKINS, Member AIME, formerly Assistant Operating Manager, Coplay Cement Manufacturing Co., Coplay, Pa., is now Project Superintendent, Freeport Sulphur Co., Carlsbad, N. M.

Discussion on this paper, TP 3387AH, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Feb. 25, 1952. New York Meeting, February 1952.

Sherman, engaged as consulting engineer to determine the feasibility of mining the limestone remaining in the quarry walls, advised that there appeared to be large reserves of mineable limestone and recommended opening a level on the lowest quarry floor to prospect and develop the exposed beds by trackless equipment.

In December 1949, 100 ft of experimental drift were driven with a truck-mounted jumbo, a front-end loader, and 5-yd quarry trucks. The program indicated that the optimum size of heading was 24 ft wide and 12 ft high, this heading width requiring no support and the back being low enough to be easily scaled and inspected. The folding made both breaking and scaling more difficult than that usually encountered in flat-bedded deposits, but drifting indicated that rock could be produced, and at a lower development cost than for outside sources of limestone. It was decided to purchase equipment and proceed with a development program.

A reconditioned Joy 16HR, 440-v ac loader with a gooseneck conveyor for truck loading was purchased. A Joy 60E12 diesel-electric shuttle car was ordered and arrangements were made to use 5-yd trucks until it was delivered. A truck jumbo equipped with two hydro-drill jibs with 6-ft chain feeds and 3½-in. drifters was on hand. Seven-ton skips hoisting in balance to the crusher on the cement rock level were available from the previous quarrying. The rock was to be blended with the cement rock as evenly as possible at the crusher. About 1000 tons of cement rock were crushed per day; experience had indicated that this allowed 300 tons of limestone per day to be hoisted from the mine.

The development headings were laid out to follow the limestone beds exposed in the quarry walls. To maintain the highest possible grade, strike fault zones and dolomite beds were left in pillars and crosscut only where needed for access and ventilation. The structure was rolling and plunging and it was necessary to follow the contacts closely to break clean limestone. These proved to be too obscure to follow by eye and required sampling each round. Results are posted on 20-scale sample maps.

Mine personnel were selected under the procedure set forth in the contract, the union requiring the classification and posting of all jobs for bidding. Awards were made to the applicants on the basis of seniority, eliminating the possibility of selection or training by promotion. None of the applicants had had any previous mining or quarrying experience. It was apparent that the success of the venture depended on developing simple and safe methods for each job and training the men in their use. The first step was to develop a standardized drilling and blasting method.

Experimental headings had been broken with 12-ft holes drilled in a V pattern. A three-man crew required two shifts to drill and blast, using a dual jib jumbo. The average round, requiring 1.8 lb of powder per ton, broke 160 tons; the average advance was slightly over 6 ft and the efficiency was about 55 pct. A study disclosed that: 1—the men could not properly point the 12-ft holes; 2—where the cut holes were properly drilled the side holes would bootleg from 1½ to 2 ft; 3—the 60 pct dynamite was too fast for the rock; 4—changing steel should be eliminated wherever possible; and 5—the dual jib jumbo was unstable.

The round was shortened to 10-ft cut and 7-ft side holes and the jumbos changed to single jibs. This eliminated 75 pct of the steel changing. A 10-ft pipe with guide bars welded at the cuthole angles centered on the floor at the face was used as a template by the drillers. Five parallel flatback holes were used to eliminate a line of weakness in the back caused by drilling the V cuts all the way up the face. To stimulate the miners' interest, time was taken to discuss each round after blasting. Within a month the drillers had achieved a fair degree of skill in pointing their holes and properly timing the rounds. At this time the company introduced 40 pct Atlas Giant gelatin with a 90-stick count and Rockmaster millisecond delays. An improvement in breaking was noted. The performance when this shorter round was used was 130 tons per round, a powder consumption of 1.3 lb per ton, an advance of 5.5 ft per round, and efficiency of 77 pct. The two-man crew drilled and blasted in two shifts.

It was now felt that the drillers had attained enough skill to permit reduction of the 2-in. diam hole then being used. There was an improvement in drill speed and powder consumption when 1⅝-in. single pass bits were used. Before long, however, there was difficulty with chipped wings and bits lost in the hole, and a change was made to threaded tungsten carbide bits. Because of thread trouble, the company adopted and still uses Rockbit Intraset, with carbides integrally set in alloy steel rods.

To take advantage of the increased drilling speed the round was lengthened by 12-ft cut and 9-ft side holes. Chain feeds were extended to 9-ft runs. This 40-hole round averages 188 tons, an advance of 8 ft, with powder consumption of 0.97 lb per ton and an efficiency of 87 pct. Two men drill and blast two rounds in adjacent headings in three shifts. Part of the reduction in powder consumption is due to the use of three sticks of gelatin in the bottom of each hole with the primer, the balance being gelodyn No. 3, which has a stick count of 120.

Three driller blaster crews now break 300 tons per day besides doing the extra scaling, pipe laying, and toe drilling in the cement rock quarry. The improvement in tons per man shift from 26 to 63 and powder consumption from 1.8 to 0.97 lb per ton indicates that efforts in training the men have been well spent. A future study will determine possibilities of reducing hole diameter to 1⅜ in. and drill steel to 1⅛ in., using smaller machines.

For a year before the shuttle car was delivered, three 5-yd trucks, each hauling one skip load of 7 tons, were required to handle 300 tons per shift. The shuttle car replacing the three trucks hauls two 7½-ton skip loads per trip and there is a wait of 3 to 4 min for the second skip. The car cycle from 1000 ft one way is 12 min: 5 min total travel time, 3 min at the loader, and 4 min at the skip. The two sections of the mine are at different levels connected by grades of 15 pct, which are negotiated without difficulty. Top tonnage to date has been 435 tons from 1000 ft.

Company experience has shown that the diesel electric car combines the best features of the cable reel and the battery-type car. General specifications for this car are very similar to those for the Joy 60E rock type widely used in potash, limestone, and iron mines. The power unit is a General Motors Series 371 diesel, driving a 250-v dc generator. There are three 15-hp series-wound gear motors, two on traction and one driving the conveyor and hydraulic pump for the steering booster. A very effective electric brake is provided in addition to

hydraulic disk brakes on the rear wheels and band brakes on the front wheels. The slower application of voltage by the diesel-electric drive seems to favor the motors, drive chains, and conveyor chains. The absence of a gear shift simplifies driver training.

Hauling over grades has proved that the greatest possibility of a major breakdown is the destruction of the armature by overspeeding. A top speed of 8½ miles per hr has been set by the manufacturer and a warning device is provided. From an operating standpoint, some means of automatically cutting in the electric brake would be more satisfactory. The driver station should also be raised for better visibility in sinuous headings.

The chemical-type exhaust scrubber uses 10 lb of aqueous sodium sulphite and 0.5 lb of hydroquinone in 15 gal of water. As the exhaust gases evaporate the water the level is maintained automatically from a 45-gal makeup tank. The scrubber carries sufficient water and chemical for a full-shift operation. Irritants are not noticeable and the weekly tests of the exhaust gas show less than 0.005 pct CO. In an 8-hr shift 9 gal of fuel are used.

The loader crew consists of a loader operator, a helper, and two scalers. They do all the loading, scaling, and moving and greasing of the loader, which will be used to load from future stopes. There has been some stope preparation and surface loading in high faces to test the performance of the loader under these conditions, revealing the fact that the condition of the floor has more to do with efficiency of the loader than any other factor. As the rounds were broken with smoother floors and more even walls the loader breakdowns decreased. It has been possible to load headings driven on plus or minus 15 pct grades without difficulty.

The size of the headings and frequent breakthroughs to the quarry face have resulted in good natural ventilation in the mine. Joy 5-hp Axivane fans with 18-in. vent tubing provide additional ventilation in the most advanced headings. The mine air is tested weekly with a CO detector.

The training and safety program has removed the prejudice originally held by the employees against mining. The turnover is low and there is no difficulty in obtaining replacements. The largest safety problem at the start was to build the confidence of the men by training them to feel that if they followed safe working practices they were in no danger. For this reason the safety features of the new methods were consistently stressed rather than the efficiency. The two years without a lost time accident confirm the correctness of this approach. The two unusual hazards for inexperienced men were explosives and falling ground. Strict supervision of explosives handling and blasting practice is possible in a small operation. Loose ground is more difficult to check; each man, therefore, is trained in scaling when he starts in the mine, and it is emphasized that he is responsible for the place in which he works. Two scalers are carried on the crew to make sure all new blasts will be scaled. This is a high proportion of man hours devoted to scaling, but an early accident would seriously have jeopardized the future of the mine and the company's investment.

Table I. Operating Statistics for Trackless Equipment

Tons per day	300	Holes per round	40
Active headings	14	Tons per round	188
Jumbos	3	Lb per ton explosives	0.97
Driller-blasters	6	Tons per jumbo shift	126
Loader crew	2	Tons per driller shift	63
Scalers	2		
Shuttle car	1		
Total no. of men	11		
Tons per man hour	3.2		

Since the mine is primarily on a development basis it has not been possible to set up a panel system with a definite cycle of operations. However, it has been found economical to follow certain rules in organizing this work: 1—Headings should be driven in groups of two or more to save setting-up time for the jumbos and loaders. 2—A ratio should be maintained of 4 active headings to each jumbo. 3—All new conditions that arise should be carefully studied, methods developed, and the men carefully instructed in the mine, with the emphasis on safety.

Table I summarizes present operating statistics.

Acknowledgments

The writer wishes to acknowledge the kind permission given by M. E. Grunewald, President, and D. J. Uhle, Vice President and General Manager, to publish this paper. He wishes also to acknowledge the assistance given by A. F. Rambosek, Chief Engineer, and L. M. Yarberry, Mine Superintendent.

The Brown Iron Ore Resources of Missouri

by Edward L. Clark and Garrett A. Muilenburg

THE first record of the discovery of iron ore in Missouri was Marquette's observation in 1673 of brown iron ore, or limonite, in the Mississippi River bluffs just north of the mouth of Apple Creek in southeastern Perry County. There is no record of any attempt to mine or smelt Missouri iron ores until 1815, when the Ashebran furnace was erected at the Stouts Creek shut-in near Arcadia, Iron County. Some limonite was treated in this furnace, the first iron furnace west of Ohio, but most of the ore was hematite from Shepherd Mountain and adjacent deposits. Eight years later, in 1823, the Perry furnace five miles north of Caledonia, Washington County, was constructed to smelt limonite ore. It

E. L. CLARK, Member AIME, is State Geologist, Missouri Geological Survey, Rolla, Mo. G. A. MUILENBURG is Assistant State Geologist, Missouri Geological Survey.

Discussion on this paper, TP 3696L, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Nov. 3, 1952. Chicago Meeting, September 1952.

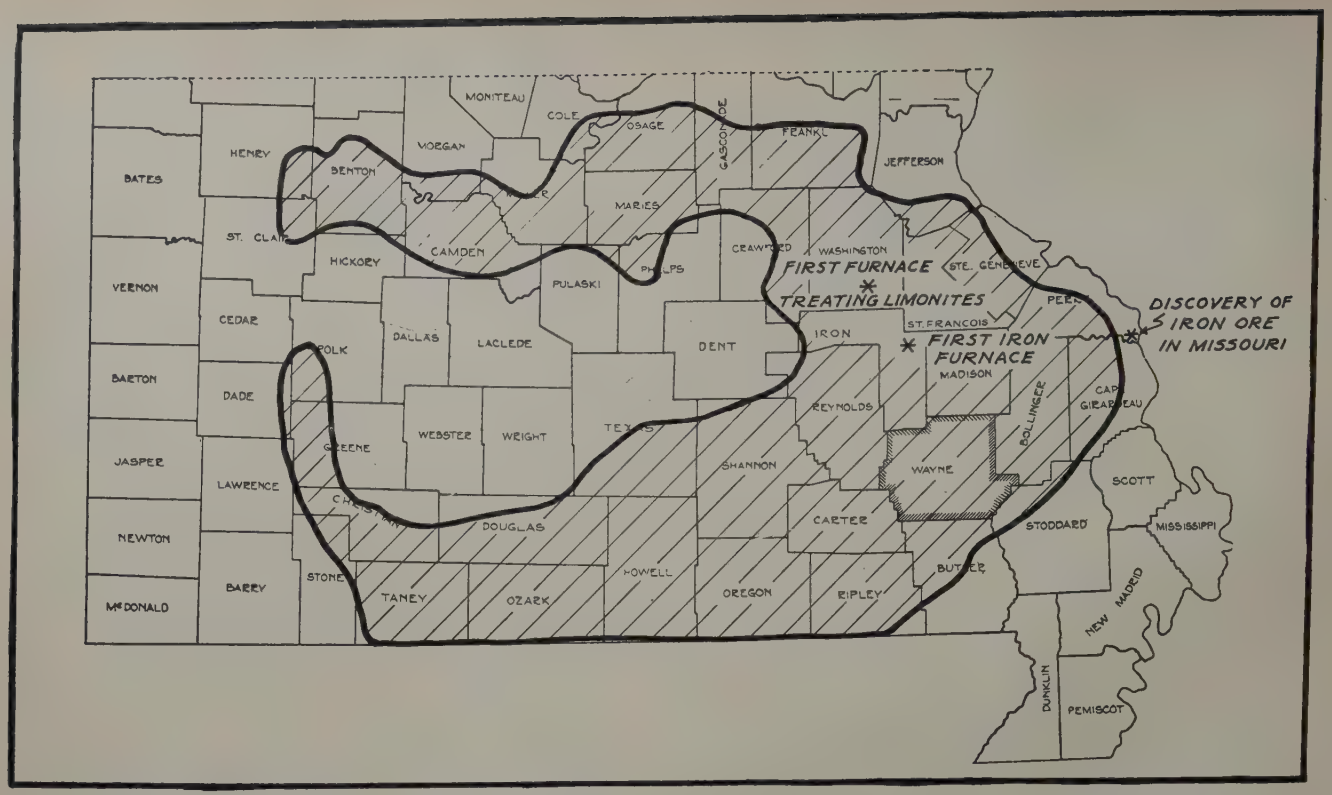


Fig. 1—Distribution of limonite ores in southern Missouri.

was not until 1890 to 1900, however, that these ores attracted considerable attention. Nearly 300,000 tons had been produced by 1911, and this production had come from 24 counties in the Ozark region. For the next two decades, until 1931, essentially all activities ceased. During the depression years of the thirties when employment was low many people turned to the brown ores for subsistence; consequently production was increased. World War II produced another stimulus to the industry. The availability of risk capital in the past three years has revived interest in these deposits, although no appreciable increase in production has resulted.

Character of the Deposits

On the basis of their possible origin, Crane[1] divided the limonites of Missouri into two classes. Deposits that had been derived by oxidation and hydration of marcasite and pyrite were termed secondary, and those precipitated directly as hydrous ferric oxide from solution, much as bog ore would be formed, were termed primary. It has been difficult for the present writers to distinguish these classes on the basis of the physical character of the ore. There is, however, a sharp distinction in mode of occurrence of the two types or classes in southeastern Missouri. The secondary limonites bear no relation to topographic conditions except that they are more frequently exposed on slopes where there has been active erosion. The primary limonites of Crane, on the other hand, are found on or near the crests of the ridges and divides adjacent to and in the Gulf Coast embayment of southeastern Missouri. In a local area the primary deposits have common elevations, with a regional dip toward the southeast.

The individual deposits, or *banks*, vary greatly in size and outline. Those associated with limestone, dolomite, and solution breccia are usually small and contain only a few tons of ore. The deposits in the residuum may be large, but with few exceptions individual deposits have not exceeded 75,000 tons. The majority of the banks are rarely more than 75 ft across and 35 ft deep, and relatively few pits have a long axis greater than 150 ft.

On the surface the exposures are attractive, and the ore appears to be all over the ground, its resistant character having caused it to concentrate in the upper soils. Because of the high specific gravity of the limonite masses the ore is left behind as lighter fractions of the residuum and bedrock are eroded away. The tendency for the ore to creep or move downslope always exaggerates the amount present. In some areas there were literally hundreds of tons of such boulders on an acre of ground. When mining got under way and these surface boulders had been recovered, the actual size of the deposit became greatly restricted, and in many instances there was no underlying orebody. The close proximity of one pit to another has often misled the prospector and miner to believe they are connected and continuous over large areas. Such interpretations are followed by the speculator or promoter.

Character of the Ores

The brown ore consists predominantly of limonite. Small amounts of hematite and goethite may be present. The gangue consists of chert fragments, clay, silica sand grains, and iron sulphides, both marcasite and pyrite. Quartz along fracture planes and vugs may be present. In many occurrences brecciated masses and individual fragments of chert are so abundant as to prohibit exploitation. The cellular porous ores usually carry much clay that must be washed out before shipment. Some large boulders of ore contain cores of unoxidized pyrite and marcasite. These sulphides normally increase in depth,

but they have been observed in large residual boulders of weathered ore on the surface.

On the basis of physical character the limonite can be divided into five classes: hard dense boulder ore, cellular or cindery boulder ore, tabular ore, stalactitic or pipe ore, and soft granular or ocherous ore. The dense boulder variety has constituted most of the production. Surface expressions of a deposit are usually the hard boulder, tabular, or stalactitic varieties of ore. The apparent light weight of the cellular boulder ore and the soft earthy character of the ocherous ore have undoubtedly caused some potential deposits to be ignored.

Botryoidal structure is prevalent in the tabular variety where it has been developed on the unfilled portion of a vein or large solution opening. Such structures have led the prospector to believe he is near the mother lode because the limonite appears to have been solidified from a molten mass.

Geological Occurrence and Geographic Distribution

Most of the production has come from the residuum from the Potosi, Eminence, Gasconade, Roubidoux, and Jefferson City formations of Cambro-Ordivician age. In southwestern Missouri the ore is related to the erosional surface between Mississippian limestone and Pennsylvanian shale or sandstone. However, small deposits are so common throughout southern Missouri that they may be expected in residuum from any stratigraphic unit between the pre-Cambrian and the Pennsylvanian. Attempts to assign occurrences in a given geographic area to a definite restricted stratigraphic zone have not been successful.

Occurrences of limonite deposits are known in every county in the Ozark region, the adjacent Springfield plain on the western slope, and the adjacent counties in the Gulf Coast embayment, see Fig. 1. Although the distribution is widespread, occurrences are concentrated in an arcuate belt that extends from west-central Missouri eastward to the southeastern part of the Ozark Mountains, then westward to the southwestern corner of the state. Scattered deposits occur in the central area, but the degree of concentration is appreciably less. Wayne, Butler, Howell, Shannon, Ripley, Oregon, Christian, Greene, Madison, and Stoddard counties have contributed approximately 90 pct of the total production.

Exploration

Most of the deposits in Missouri have been placed in production with very little if any exploration. Under these conditions production continued until the ore was depleted or had reached such a grade as to prohibit economical recovery. In brief, exploration was actually mining.

In 1943 and 1944 the U. S. Bureau of Mines examined many surface expressions of possible deposits. Nine were selected for detailed exploration.[2] Rotary-bucket drilling, power augering, test pitting, with shafts, and bulldozer trenching were employed. For the deeper exploration the rotary-bucket drill was adequate and depths of 50 ft were reached with a 16-in. bucket. The advantage of this method over other drilling methods is the large amount of sample obtained and the large size of fragments in the sample. The power-auger was able to reach depths up to 100 ft, but the great amount of chert caused excessive wear on the cutting heads and the spiral flights.

Table I shows the extent of combined exploration on the nine deposits investigated.

The ore developed by this exploration program totaled 91,000 tons averaging 37.4 pct iron. The ore reserves developed on the individual deposits ranged from 0 to 40,000 tons.

Mining and Beneficiation

The brown iron ores present a difficult problem in mining under present conditions. Inasmuch as the deposits are relatively small it has not been economically feasible to attempt large-scale production. Several deposits in a given area may be operated as a unit by providing each one with the necessary loading and hauling equipment and washing and concentrating at a central plant.

Table I. Exploration of Nine Deposits in Southeast Missouri

Type of Exploration	Holes or Cuts, No.	Depth of Penetration
Bucket-drill	105	2424 ft
Bulldozer	6	1000 cu yd
Power auger	34	1089 ft
Test pits	21	218 ft
Total	166	3731 ft 1000 cu yd

When production was at the maximum much hand labor was employed. In some mines, where conditions were suitable, power-operated scrapers and shovels were used but even when such power equipment was used much ore was hand sorted. Such methods produced a grade of ore that would assay from 54 to 56 pct iron. Hand labor is no longer available in the brown ore region and present methods are entirely mechanical. Bulldozers are used for clearing and stripping the ground and power shovels or draglines load the dirt into trucks for hauling to the concentration plant.

Concentration consists almost entirely of washing in a log washer to remove the clay, which constitutes one of the chief impurities. The mine-run ore is dumped on a grizzly usually constructed of old rails, and washed into the log washer with a stream of water from a high-pressure hose. Large chert boulders and other waste are removed from the

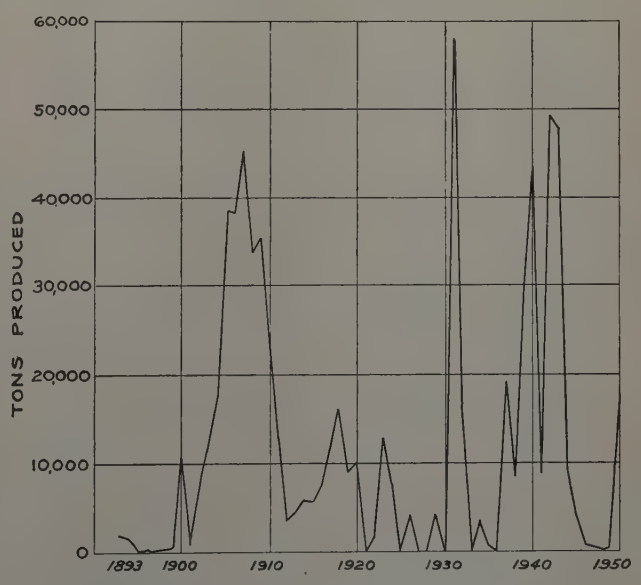


Fig. 2—Brown iron ore produced in Missouri. (After Bishop, *The Mineral Industry in Missouri in 1946 and 1947.*[3])

grizzly by hand. The overflow from the logs goes to a settling pond for removal of solids, and the cleared water is recirculated. The discharge from the head of the logs is passed over a wet screen from which the undersize material goes to the ore bin and the oversize to a crusher. Some oversize chert is picked from the conveyor before it reaches the crusher. If the ore contains too much silica to meet the minimum specifications for iron content, it must be removed. Jigs of various types have been used to remove excess chert, but no standard practice has been worked out. Differential settling in a heavy medium has not been tried in practice. A pilot plant is now using a spiral classifier for the logs.

A water supply of ample capacity and the disposal of waste are serious problems in a washing operation. The ratio of waste to ore varies considerably from one deposit to another and the size of the settling area required will be proportional to the size of the deposit and the concentration ratio.

Production

Approximately 700,000 tons of brown ore have been produced, see Table II. Although the total tonnage is small it has been significant in the economy of the areas of production. In many respects it is *poor man's ore*. When difficult times arrived, the individual farmer became an iron miner by picking up surface boulders and hauling them to the nearest railroad. During the depression years of the early thirties, before relief became an accepted policy, it was not uncommon to see small individual stock piles of 5 to 25 tons of ore at each village.

Table II. Annual Production of Brown Iron Ores in Missouri in Long Tons

Year	Brown Ores, Primary and Secondary	Year	Brown Ores, Primary and Secondary
Prior to 1893	26,015	1921	
1893	1,800	1922	1,282
1894	1,220	1923	12,966
1895		1924	7,422
1896	60	1925	
1897		1926	4,371
1898		1927	
1899	333	1928	
1900	10,454	1929	4,155
1901	292	1930	315
1902	8,520	1931	58,233
1903	11,574	1932	15,797
1904	17,642	1933	
1905	38,637	1934	3,704
1906	38,351	1935	126
1907	45,367	1936	
1908	33,632	1937	18,291
1909	35,217	1938	8,331
1910	22,542	1939	29,415
1911	12,309	1940	43,523
1912	3,399	1941	8,738
1913	4,125	1942	49,048
1914	5,500	1943	47,715
1915	5,145	1944	9,050
1916	7,346	1945	4,000
1917	11,393	1946	536
1918	16,013	1947	265
1919	8,759	1948	
1920	9,671	1949	150
		1950	17,500
		Total	720,249

Table II gives the total reported production of limonite that has been shipped to consumers. With shipments being made by scores of individuals, it is not possible to obtain an accurate figure, but figures reported in Table II are believed to be within 10 pct of the total amount that has been mined.

The production of limonite ore has been activated by economic depressions and World Wars I and II. When economic conditions have been good and there was no national crisis or artificial stimulus, production sharply declined, Fig. 2.

Reserves and Future Potential

For the purpose of the following discussion, ore is considered to be material of not less than 35 pct iron, in a physical condition requiring a minimum of concentration to produce a marketable product. Standards for marketable concentrates in the past have been not less than 50 pct iron, dry basis, and not more than 10 pct silica and 0.2 pct sulphur.

Under such standards there are less than 100,000 tons of proved limonite ore reserves in Missouri. The lack of reserves may be attributed to existing exploration and development practices. The character of the individual deposits is not conducive to large exploration or development programs that calculate ore reserves in advance of production. On the other hand, when hundreds of known occurrences are given consideration the inferred ore reserves exceed the total of past production.

Although the total potential reserves are large, they will through necessity be obtained from many individual deposits that may contain no more than 5000 to 25,000 tons. Production schedules will be difficult to establish and maintain unless an organization is operating several deposits simultaneously. There does not appear to be a sufficient reserve available to sustain ferrous metallurgical industries of any appreciable size, especially if such industries are entirely dependent upon local raw materials. It appears more likely that these ores will continue to find their way to markets that need high-quality iron ore low in silica, sulphur, and phosphorous.

New beneficiation processes will be needed to permit mechanization of the mining of these ores. Until such a process is developed, there will be continued waste of potential ore by inefficient treatment, the shipment of a lower grade of concentrate, and the retardation of the development of many potential deposits. A central washing plant treating ore from several mines will permit exploitation that is not realized today. Likewise production can be increased by a program of subsidizing independent contractors with private capital, equipment, and mining experience, followed by a fixed base price on ores shipped, with adjustment of prices after returns have been obtained from the furnace or consumer. Such a program will release the independent operator from the somewhat unsatisfactory treatment he has experienced in the past because he could not maintain a constant grade or quantity of shipping ore.

Although the grade of limonite concentrates produced in Missouri is in demand on the open market, and the potential reserves are large, no large increase in annual production is expected soon.

References

[1] G. W. Crane: The Iron Ores of Missouri, Missouri Bur. Geol. and Mines, 2nd ser., **10**, p. 54, 1912.

[2] H. J. Ballinger and P. Pesonen: Investigations of Southeast Missouri Secondary Limonite Deposits, U. S. Bur. Mines R. I. 4314 (1948) pp. 6-7.

O. M. Bishop: The Mineral Industry of Missouri in 1946 and 1947 with Total Production Summarized. Missouri Geol. Survey *I.C. 4* (1949) p. 39.

Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals

by Shiou-Chuan Sun

THE fact that coals of different ranks and even of the same rank differ greatly in their amenability to froth flotation is well known. In recognition of the need for an explanation of this phenomenon, two hypotheses have been suggested. Wilkins[1] reported that the floatability of coals increased with an increase of the carbon content or rank. This postulate is handicapped by the fact that bituminous coals that possess moderate carbon contents are actually more floatable than anthracite coals that have high carbon contents, as shown in columns 6 and 9 of Table I. Taggart and his associates[2] implied that the difference of floatability between bituminous and anthracite coal was caused by the variation of carbon-hydrogen ratio. This is not applicable to the relative floatability of other coals and carbons. For example, column 11 of Table I shows that the carbon-hydrogen ratios of low-floating lignitic coal and non-floating animal charcoal are not only smaller than the moderate-floating anthracite coal, but are also similar to the high-floating bituminous coal. Furthermore, according to this hypothesis, high temperature coke-A (464), Ceylon graphite (1238), and lamp-black (357), all possessing extremely high carbon-hydrogen ratios, should be less floatable than other substances having much lower carbon-hydrogen ratios such as high volatile-B bituminous coal (11.9 to 22), anthracite coal (35.7 to 60.5), lignitic coal (15.6 to 33.6), and charcoal (13 to 26.2). However the former group is actually more floatable than the latter group.

In this paper, a *surface components hypothesis* is proposed to explain the different floatabilities of coals, carbons, and hydrocarbon minerals. The validity of the hypothesis is experimentally supported by the actual floatability, natural floatability, wettability, and adsorbability for neutral oils of coals, carbons, and hydrocarbon minerals tested.

The combustible recovery of the flotation results, as used in this paper, was calculated from Eq. 1:

$$R_c = \frac{P\ (100-E_p)\ \ 100}{F\ (100-E_f)} = \frac{R_w C_p}{C_f} \qquad [1]$$

where R_c is the percent combustible recovery; F and P are, respectively, the weight of feed and the weight of concentrate or product; E_f and E_p are, respectively, the total percent of ash plus moisture in feed and in concentrate; R_w is the percent weight recovery; and C_f and C_p are, respectively, the percent of combustible in feed and in concentrate. Except for ash and moisture content, all chemical components of a coal are assumed combustible.

S.C.SUN, Member AIME, is Associate Professor of Mineral Preparation, the Pennsylvania State University, State College, Pa.

Discussion on this paper, TP 3615F, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Nov. 24, 1952.

The experimental work included studies on flotation, ultimate and proximate analyses, contact angle tests, extractions of bitumen-A with benzene, adsorptions for liquid hydrocarbons, and wetting tests. Most of the flotation experiments were performed in a laboratory Fagergren machine; others were tested in a small Denver machine. The solid feed for the former was 300 g and for the latter was 30 g. The solid materials used for flotation were crushed to —48 mesh. After the mineral pulp in the flotation cell was agitated for 6 min and the pH was adjusted to 7.5 ± 0.2 with sodium hydroxide or hydrochloric acid, a petroleum light oil having a viscosity of 5.73 centipoises at 77°F was added and conditioned for 2 min. Finally, pine oil was introduced and the froth was collected for exactly 3 min. The weight ratio of petroleum light oil to pine oil was kept constant at 1.5 to 1. Tap water was used for all flotation tests.

Contact angles were measured with a captive bubble machine. For each coal sample, three specimens were mounted in transoptic mounts and polished with levigated alumina, first on a sheet glass, then on a cloth-covered metal polishing wheel. The polished specimen was first washed with distilled water and wiped thoroughly on a cleaned linen pad, then transferred into the pyrex cell of the captive bubble machine and conditioned for 6 min., and finally measured for contact angles at three or more points. Except where otherwise stated, the induction time for each measurement was 1 min. The contact angle representing each material was obtained by averaging the measurements of three specimens. The linen pad was first washed with warm distilled water, then boiled 30 min in a 2N sodium hydroxide solution, and finally washed with distilled water until no trace of sodium hydroxide could be detected in the decanted solution. The cleaned linen pad was stored under distilled water. Immediately before using, the pad was rewashed and transferred into a clean pyrex petri dish partly filled with distilled water. The glassware and rubber gloves used were cleaned by soaking in sulphuric acid-potassium dichromate cleaning solution, followed by rinsing with distilled water. The polished specimens were handled only by glass forceps.

The ultimate and proximate analyses were made in accordance with the ASTM standard procedures for coal and coke. The extractable bitumen-A was determined by weighing 1 g of —100 mesh sample and placing it in a desiccated and weighed ASTM aluminum-extraction thimble. The thimble was placed in condenser hooks and inserted into an extraction flask containing 100 cu cm of benzene. The flask was heated and the benzene vapor was condensed by water coils. At the end of 24 hr of percolation, the thimble was removed, desiccated, and weighed. Loss in weight of sample was taken as bitumen-A and calculated to dry and ash-free basis.

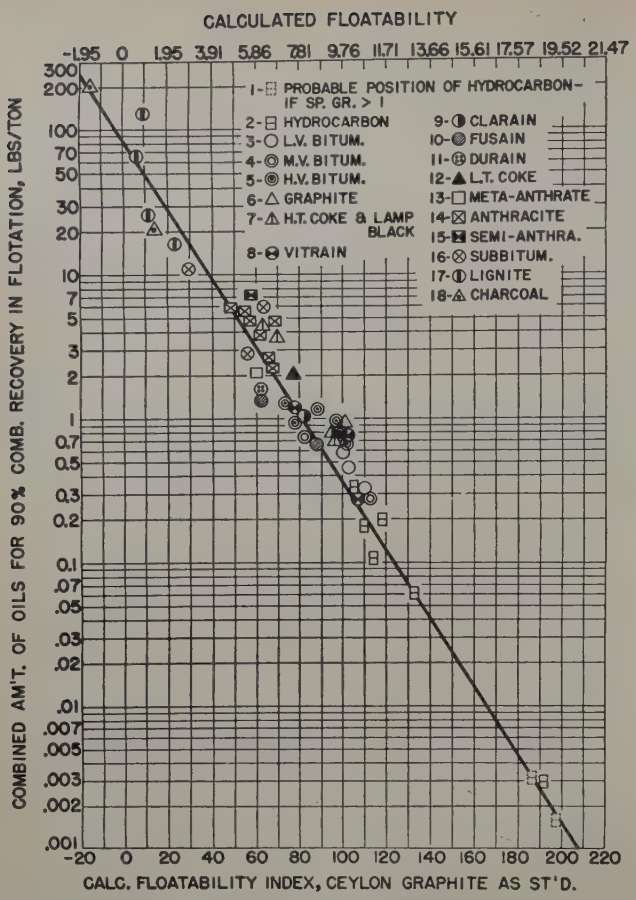


Fig. 1—Correlation between the actual floatability and the calculated floatability index of coals, cokes, and hydrocarbon minerals.

The adsorbability of materials for liquid hydrocarbons was determined according to the following steps: 1—The sized 48x65 mesh material was washed, dried, and stored in a clean desiccator. 2—A 5-g sample was accurately weighed and transferred into a cassia flask having a graduated neck and a ground-glass stopper. 3—After the addition of 40 cc of distilled water, the flask was shaken for 20 min and allowed to stand for 12 hr. 4—Five cubic centimeters of liquid hydrocarbon were added and the flask was shaken for 10 min. 5—The mineral pulp was transferred into a pyrex fritted Gooch crucible and filtered for 1 hr by a water aspirater. 6—The sample, together with the Gooch crucible, was desiccated for a predetermined time and weighed. 7—The difference between the weight of the hydrocarbon-treated sample and that of the original sample was calculated and considered to be the amount of liquid hydrocarbon adsorbed by the sample. 8—The result was further checked by means of ether extraction of liquid hydrocarbon from the filtrate. The time required for desiccation was predetermined by the same procedure given in steps 1 to 6 except that liquid hydrocarbon was omitted, and the sample was kept in a desiccator and weighed after each 20-min interval until its original weight was reached. The amount of liquid hydrocarbons adsorbed by the crucible itself was also predetermined, and the data were taken into account during the calculations of step 7. Obviously the analytical results are only approximately correct.

The wettability of various minerals in distilled water was determined by using 48x65 mesh quartz as a standard particle size. The equal-settling particle sizes[3] of other minerals were calculated and prepared by screening. The sized particles were thoroughly cleaned, washed, dried, and stored in clean glass jars. For each test, 1 g of the sample was spread on the surface of 250-cu cm distilled water in a 400-cu cm pyrex beaker. The water was then stirred gently by a glass rod rotating at a constant speed of 100 rpm. The time required for approximately 95 pct of particles to fall below the water surface was taken as a measure of its wettability.

Surface Components Hypothesis

A surface components hypothesis is proposed to explain the different floatabilities of various coals, carbons, and hydrocarbon minerals. The gist of the hypothesis is that these heterogeneous materials consist of both floatable and non-floatable chemical constituents and that their floatabilities are governed by the balance between these two groups of components. The surface of one material predominating with floatable components is more floatable than that of another material predominating with non-floatable components. The floatable components are oil-avid and water-repellent, whereas the non-floatable components are water-avid and oil-repellent.

Natural coal is generally believed to be a chemical and physical mixture of hydrogen, carbon, sulphur, oxygen, moisture, mineral matter, and nitrogen. The flotation properties of these components can be roughly estimated from the test data of Tables I, III, and IV. For example, the hydrogen content of coal on dry and ash-free basis is believed to be tied up in the form of hydrocarbon[4, 5, 6a, 7] and should be treated as such. Tables III and IV show that hydrocarbons, as represented by wood rosin and beeswax, are water-repellent and oil-avid and consequently should be highly floatable. This is proved to be true by the test data of Table I. In a similar way crystalline carbon, represented by Ceylon graphite and purified spectrographic electrode, is estimated to be moderately floatable; and sulphur,[6b] represented by an average of the floatable element sulphur and the non-floatable pyrite, is considered to be slightly floatable. The unfloatability of moisture, oxygen, and mineral matter can be visualized from Tables I and IV. Table I shows that the non-floatable lignite and animal charcoal are characterized by their high contents of moisture, oxygen, and mineral matter. The deleterious effect of oxygen on the floatability of coals and coke has been described in a separate treatise.[8] Table IV shows that the adsorbability for kerosene of lignite, oxidized coal, and mineral matter, which is represented by slate, calcite, pyrite, and quartz, is extremely low as compared with other tested materials. Owing to the lack of sufficient information,[6c, 9] nitrogen is tentatively considered as being slightly non-floatable. Tests 13-16 of Table III show that the contact angle of a high purity graphite stick decreases slightly after being nitrogenated at 1100°C for 5 hr and then cooled to room temperature in the porcelain tube of a Globar furnace. Throughout the heating and cooling a stream of purified nitrogen gas was passed through the tube. A detailed description of the furnace and procedure for making nitrogenous graphite has been reported by Walker.[10] Judging from these tests and the uniformly low content of nitrogen in the tested coals, the role played by nitrogen is relatively unimportant as compared with other components. The above classification of the flotation

Table I. The Ultimate Chemical Analysis, Floatability Index, Actual Floatability, Carbon-Hydrogen Ratio, Contact Angle, Extraction of Bitumen A, Calorific Value, and Specific Gravity of Coals, Carbons, and Hydrocarbons

Test No.	Sample	Ultimate Analysis, Pct: As-Received Basis H_2O (1)	As-Received Basis Ash (2)	Dry and Ash-Free Basis O (3)	N (4)	S (5)	C (6)	H (7)	Calculated Floatability (8)	Calculated Floatability Index (Using Ceylon Graphite As Standard) (9)	Combined Amount of Petroleum Light Oil and Pine Oil for 90 Pct Comb. Rec. in Flotation, Lb per Ton (10)	Carbon-hydrogen Ratio, Dry and Ash-Free (11)	Average Contact Angle in Distilled Water (12)	Extraction of Bitumen-A with Benzene, Pct Dry and Ash-Free (13)	Sp Gr (14)
1	Petroleum Light Oil, Bradford, Pa.	0	0	0.49	0	0.01	85.4	14.1	23.83	243.2	na.f.	6.1			0.82
2	Kerosene	0	0	0.8	0	0.08	85.5	13.61	23.09	235.6	na.f.	6.3			0.72
3	Pure White Bees-wax, E. & A. Co.	0.09	0.02	6.17	0.04	0	80.2	13.4	21.36	217.9	na.f.	6.0	109		<1
4	American Montan Wax-c	—	0.03	7.95	0	0.3	79.5	12.25	19.38	197.7	na.f.	6.5	105		<1
5	Uintahite (Gilsonite), Utah	0.3	0.19	1.15	2.11	0.52	85.4	10.51	18.48	188.6	0.003[b]	8.1	90	99.9	1.07
6	Wurtzilite, Utah	0.62	0.69	2.46	1.93	3.75	80.6	10.68	18.16	185.3	na.f.	7.6			<1
7	Wood Rosin-K, Hercules	0	0	17.44	—	0.06	73.5	9.0	12.52	127.8	0.06[b]	8.2	70		1.07
8	Resin-600°C, Allied Chem. & Dye	0.1	0.2	1.212	1.5	0.71	91.6	5.0	11.19	114.2	0.10[b]	18.3	50	93.0	1.11
9	Hard Pitch, Allied Chem. & Dye	0	0.1	1.2	1.0	0.5	93.0	4.1	10.05	102.5	0.17[b]	22.7	39	43.0	1.23
10	Tasmanite, Australia	0.8	60.92	7.97	0	4.54	75.0	10.48	11.51	117.5	0.20[b]	7.2	51	3.9	1.36
11	Vitrain (m.v.), Springdale, Pa.	0.8	5.29	3.67	1.39	0.91	88.3	5.12	10.51	107.2	0.29[b]	17.2	49	0.2	1.23
12	M.v. bitum., Hastings, Pa.	0.63	6.3	4.34	1.5	0.88	88.3	5.29	10.64	108.6	0.31	16.7	62	u.d.	1.25
13	Torbanite, New South Wales	0.3	14.77	6.41	0.94	0.78	83.2	8.33	10.54	107.6	0.32[b]	8.2	57	1.2	1.19
14	L.v. bitum., Bedford, Pa.	0.35	8.0	1.19	1.56	1.77	90.9	4.58	10.51	107.2	0.32	19.8		tr.	1.3
15	L.v. bitum., Broad Top, Pa.	0.3	3.0	2.62	1.35	0.91	90.4	4.72	10.38	105.9	0.34	19.1	63	0.2	1.27
16	L.v. bitum., Windber, Pa.	0.6	7.8	2.92	1.58	1.36	89.6	4.54	9.96	101.6	0.47	19.8		tr.	1.32
17	L.v. bitum., Cresson, Pa.	0.6	5.2	3.94	1.43	0.61	89.4	4.62	9.85	100.5	0.65	19.4	60	u.d.	1.28
18	Fusain (m.v.), Springdale, Pa.	0.6	2.57	1.58	0.72	0.87	93.2	3.20	8.61	87.9	0.67[b]	37.3	24	0.5	1.38
19	H.v.-A bitum., Boone County, W. Va.	0.88	7.5	5.49	1.42	0.67	86.2	5.26	10.19	104.0	0.68	15.9		0.5	1.21
20	H.t. coke-A, Phila., Pa.	0.04	10.0	1.54	0	0.65	97.6	0.21	9.61	98.1	0.72	464.0			1.73
21	Lamp black (Martin), Spear Carbon	0.14	0.14	2.07	0.03	1.13	96.5	0.27	9.49	96.9	0.75	357.0		1.2	1.51
22	M.v. bitum., Sabinas, Mexico	0.38	22.2	4.86	1.42	2.14	86.3	5.28	7.95	81.1	0.75	16.3	45	u.d.	1.38
23	H.v.-B bitum., Stockton, Utah[a]	2.0	8.8	8.79	1.33	1.01	82.0	6.87	11.27	114.8	0.78	11.9	39	8.6	1.21
24	Vitrain (h.v.-A), Pikeville, Ky.	1.6	4.84	5.74	1.39	0.76	85.0	5.37	10.08	102.9	0.82[b]	15.8	55	0.2	1.26
25	Graphite, Colombo, Ceylon	0.03	2.18	0.83	0.06	0.11	99.0	0.08	9.80	100.0	0.90	1238.0	38	u.d.	2.21
26	H.v.-B bitum., Clariton, Pa.	1.1	8.47	6.53	1.44	1.31	83.9	5.18	9.81	100.1	0.91[b]	15.7		u.d.	1.37
27	H.v.-A bitum., Richmond, W. Va.	1.22	14.57	2.79	1.62	1.52	87.2	5.54	8.46	86.3	0.94	15.7	35	0.5	1.25
28	Clarain (h.v.-B), Ill.	3.0	4.1	12.13	1.29	2.53	78.5	5.24	7.92	80.8	1.08	15.0	45	2.7	1.24
29	H.v.-B bitum., Hopkins, W. Ky.	1.8	6.14	11.25	1.49	3.48	78.4	5.38	8.57	87.5	1.19	14.5		1.3	1.22
30	Vitrain (h.v.-B), Ill.	3.0	4.5	12.2	1.19	3.71	78.1	4.94	7.49	76.4	1.20	15.8	41	2.9	1.21
31	H.v.-B bitum., Panhandle, Pa.	1.1	8.6	14.58	1.67	1.93	77.0	4.82	7.13	72.8	1.26	19.4	37	1.3	1.32
32	Fusain (h.v.-B), Ill.	0.6	26.5	8.2	0.27	2.51	85.3	3.88	5.94	60.6	1.35	22.0	c.s.	u.d.	1.57
33	Durain (h.v.-B), Ill.	2.2	16.2	11.67	1.23	1.84	80.3	4.84	5.46	55.7	1.50	16.6	18	2.8	1.40
34	L.t. coke (prepared at 500°C)	1.66	8.9	2.63	1.56	0.72	89.9	3.37	8.16	83.2	1.7[b]	26.6		u.d.	1.32
35	Meta-anthracite, Newport, R.I.	0.1	32.2	3.56	0.15	0.16	95.8	0.29	8.78	89.6	1.8	330.0	c.s.	u.d.	2.33
36	Anthracite, Yunnan, China	0.6	0.2	1.39	0.71	0.47	95.6	1.57	6.50	66.3	2.2	60.5	50	1.3	1.69
37	Anthracite, Jeddo, Pa.	0.9	6.6	3.36	0.85	0.61	93.1	2.08	6.59	67.2	2.5	44.8	37	0.1	1.46
38	Subbitum.-B, Sheridan, Wyo.	14.2	6.0	15.65	1.74	0.68	77.4	4.53	4.38	44.7	3.6	17.1	15	11.1	1.27
39	H.t. coke-B, Clairton, Pa.	0.21	11.8	0.22	1.02	1.14	97.0	0.33	6.67	68.1	3.7	294.0		0.2	1.66
40	Anthracite, Nesquehoning, Pa.	2.2	9.0	2.42	0.79	0.61	94.1	1.98	5.95	60.7	3.95	47.8	29	1.3	1.46
41	H.t. coke-C, Johnstown, Pa.	0.36	13.1	2.91	0.02	1.23	95.2	0.64	6.19	63.2	4.3[b]	148.8		0.1	1.59
42	Anthracite, Plymouth, Pa.	1.0	9.1	3.56	1.0	0.97	91.9	2.57	6.53	66.6	4.4	35.7	30	u.d.	1.45
43	Anthracite, Eckley, Pa.	2.3	10.6	3.45	0.80	0.59	93.2	1.96	5.63	57.5	4.8	47.5	c.v.s.	u.d.	1.54
44	Anthracite bone, Jeddo, Pa.	2.0	18.8	3.82	0.76	0.59	93.0	2.0	5.17	52.8	5.5	46.5			1.65
45	Anthracite bone, Nesquehoning, Pa.	1.1	31.7	4.51	1.19	0.59	91.6	2.2	5.31	54.2	5.7	41.6			1.59
46	Subbitum.-C, Hanna, Wyo.	9.7	7.0	15.7	1.4	0.5	76.8	5.58	6.01	61.3	6.0	13.8	c.v.s.		1.24
47	Semi-anthracite, Limena, Peru	2.3	30.9	6.03	0.58	0.81	91.9	0.68	4.87	49.7	6.4	135.0	c.v.s.	1.0	2.20
48	Subbitum.-C, Gillette, Wyo.	21.7	6.2	20.27	0.97	0.98	72.6	5.18	2.88	29.4	10.6	14.0	0	28.2	1.23
49	Lignite, Velva, N. Dak.	17.5	8.6	19.37	1.18	0.46	75.0	3.99	2.20	22.5	16.0	18.8	0	5.7	1.21
50	Wood charcoal, E. & A. Co.	4.3	5.1	6.4	0.55	0.20	84.9	3.23	4.21	42.9	20.7	26.2		0.6	1.40
51	Lignite, Minot, N. Dak.	18.8	12.5	22.59	1.06	0.58	71.2	4.57	0.83	8.5	25.0	15.6	0	24.9	1.31
52	Lignite, Noonan, N. Dak.	25.9	8.0	20.88	1.26	0.86	72.4	4.60	1.17	11.9	69.0	15.7	0	37.6	1.32
53	Lignite, Dickinson, N. Dak.	34.6	5.7	7.01	1.26	0.70	88.4	2.63	0.75	7.7	130.0	33.6	0	14.7	1.20
54	Animal charcoal, E. & A. Co.	0.7	84.3	31.2	6.0	—	54.0	4.14	−1.73	−17.7	205.0	13.0	0	13.9	2.24

a, coal contains 5.36 pct of fossil resin.
b, flotation tests were performed in a 50-gm Denver flotation machine.
c, cling; c.s., cling slightly; c.v.s., cling very slightly.
na.f., natural floater; tr., trace.
u.d., undeterminable.

properties of surface components is valid only when a material is floated with an oily collector devoid of any reactive group[8, 11] and a frother, or with a frother only.

The surface components of coals, carbons, and hydrocarbon minerals cannot be identified directly by any known method and must be assessed from the analysis of their bulks. Obviously the analytical method is applicable only when the chemical composition of the surface and bulk of a material is roughly the same. This limitation does not necessarily reflect errors in the general principle of the hypothesis. Mathematically, the hypothesis is expressed as follows:

$$Fc = 3.5(CH'_{2.0796}) + y(C') + 0.4(S') - 4(M') - 3.4(O') - (N') - A$$

or in greater detail,

$$Fc = 3.5\left(\frac{H}{2.0796}\right) + y\left(\frac{C}{12} - \frac{H}{2.0796}\right) + 0.4\left(\frac{S}{32.06}\right) - z\left(\frac{M}{18}\right) - 3.4\left(\frac{O}{16}\right) - p\left(\frac{H}{2.0796}\right) - \left(\frac{N}{14}\right)$$

or in a condensed form,

$$Fc = x\left(\frac{H}{2.0796}\right) + y\left(\frac{C}{12} - \frac{H}{2.0796}\right) + 0.4\left(\frac{S}{32.06}\right) - z\left(\frac{M}{18}\right) - 3.4\left(\frac{O}{16}\right) - \left(\frac{N}{14}\right) \quad [2]$$

in which Fc is the calculated floatability representing the balance between the floatable and the non-floatable surface components; $CH'_{2.0796}$ is the relative number of the hydrocarbon molecules based on $C_{26}H_{54}$, which was reported by Warth[7] as the average paraffin wax from German brown coal; C' is the relative number of the excess or remnant carbon atoms, after the others have combined with hydrogen atoms to form $CH_{2.0796}$ molecules; S', O', and N' are respectively the relative number of sulphur, oxygen, and nitrogen atoms; and M' is the relative number of water molecules. These were all calculated on dry and ash-free basis, with the exception that water molecules were calculated on as-received basis. The symbols H, C, S, N, O, and M are respectively the weight percent of hydrogen, carbon, sulphur, nitrogen, oxygen, and moisture of the ultimate analysis as shown in Table I.

The assumed numerical values of factors x, p, y, and z for Eq. 2 are given in Table II. The variation of the value of factors x and p with the ash content of the tested materials is caused by the fact that ash tends to tarnish the floatable components, particularly hydrocarbons. The influence of ash, as represented first by a symbol A and then by $p(H/2.0796)$ in the derivation of Eq. 2 is taken into account by the factor x of hydrocarbon. The fact that the factor y of remnant carbon increases with a decrease of the hydrogen content of the tested materials may be explained by the work of Blayden and his associates.[12] They reported that the advancement of carbonization and/or graphitization of coals, cellulose, lignin, and glycine resulted not only in the increase of crystallinity and the crystal growth of their atoms, but also in the decrease of their hydrocarbon content. This led to the speculation that the floatability of carbon is directly proportional to the magnitude of its crystallinity. An explanation for the different values of the factor z of moisture is that the floatability of a relatively dry material is sharply lowered with the increase of certain amounts of moisture and that further increases of moisture to the already moistured material result in less depression.

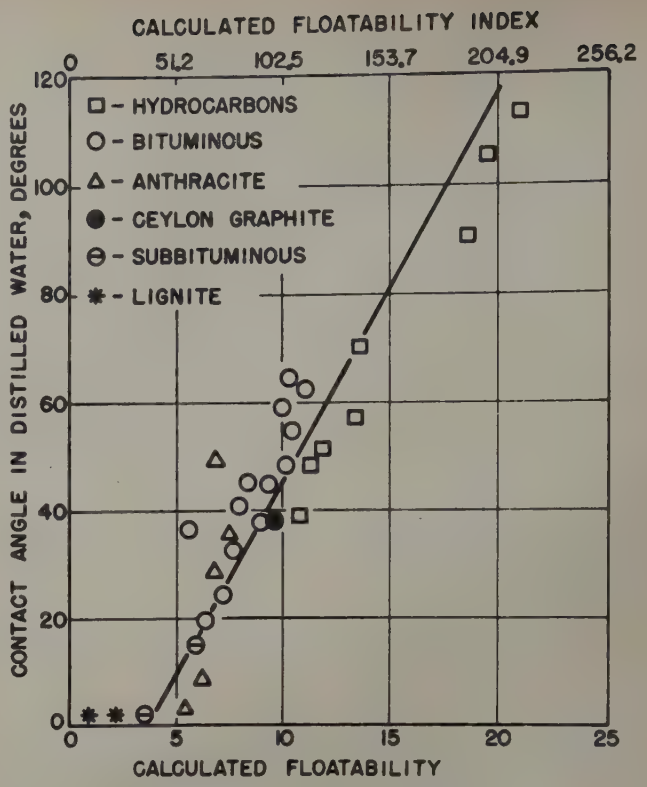


Fig. 2—Correlation between the natural floatability and the calculated floatability index of natural minerals.

Table II. The Assumed Numerical Values of Factors x, p, y, and z for Eq. 2

Ash			Remnant Carbon		Moisture	
Ash Pct, As-Received	x	p	Hydrogen Pct, Dry and Ash-Free	y	Moisture Pct, As-Received	z
0 to 8.9	3.5	0	0.08 to 0.28	1.2	0 to 14.1	4
9 to 13.0	3.0	0.5	0.29 to 1	0.8	>14.1	3
>13.0	2.5	1.0	>1	0.6		
			Porous charcoal	0.2		

The calculated floatabilities of the tested materials were converted into calculated floatability indices, using Ceylon graphite as a standard. This was done by means of Eq. 3

$$FI_c = \left(\frac{Fcm}{Fc}\right) 100 \quad [3]$$

in which FI_c is the calculated floatability index of a tested material and Fcm and Fc are, respectively, the calculated floatability of the material and the standard. By means of Eq. 2 and 3 the surface conditions and consequently the flotation properties of the tested materials can be roughly estimated. A small calculated floatability index signifies that the surface is dominated by non-floatable components and should be hydrophilic, whereas a large index denotes that the surface is dominated by floatable components and thus becomes hydrophobic.

Actual Floatability

The validity of the surface components hypothesis was checked by plotting the floatation results of the tested materials against their calculated floatability indices on a logarithmic paper and proved to be satisfactory. Fig. 1 shows that the actual floatabilities of these materials are roughly in direct proportion with their calculated indices. This relationship is mathematically expressed by Eq. 4

$$\text{Log } T = k + m(FIc) \qquad [4]$$

in which T is the combined amount of petroleum light oil and pine oil, at a constant weight ratio of 1. 5 to 1, in pounds per ton used for a 90 pct combustible recovery in actual flotation; k is the ordinate intercept at zero abscissa; m is the slope with minus sign; and FIc is the calculated floatability index.

The hypothesis is further supported by its capacity in explaining those flotation phenomena which contradict previous theories.[1, 2] For example, according to this hypothesis, the surface of bituminous coal of medium or low volatility is more dominated by floatable components than is anthracite coal, as indicated by their calculated indices. Consequently the former should be more floatable than the latter, as found in actual flotation. The small calculated index of lignite and animal charcoal indicates that the surface of these materials is dominated by non-floatable components and should result in low floatability, as evidenced by actual flotation. The analytical data in Table I indicate that the floatable components of lignite and animal charcoal are nullified by their non-floatable components, thereby resulting in extremely small calculated indices. The remaining flotation phenomena can be similarly explained.

Natural Floatability

The premise that the floatable components are much less water-avid than the non-floatable components was verified by measuring the contact angle of materials in presence of distilled water alone. Fig. 2 shows that the calculated index required for a material to exhibit a clinging contact angle in distilled water is around 65. Beyond that the contact angle generally increases with an increase of the calculated index. This indicates clearly that the surface of naturally floatable minerals is considerably dominated by floatable components and thus becomes non-polar for air-bubble contact to occur. This type of surface occurs naturally in hydrocarbon minerals, sulphur, graphite, and some of the bituminous and anthracite coals.

Tests were also made to ascertain the effect of soaking in distilled water on the natural floatability as well as on actual floatability of some natural floaters. Curves 1 to 5 of Fig. 3 show that the air-avidity of the natural floaters tested in distilled water decreases as the soaking time is increased, owing to the adsorption of water molecules and hydroxyl ions. It is significant that a material with a relatively small index requires a much shorter soaking time in reducing its natural floatability to a negligible value than another material with a large index. Curves 2A and 2B of Fig. 3 indicate that during the preliminary soaking period the adsorbed molecules and ions are only loosely attached to the surface of natural floaters and can be easily displaced by an air bubble. The displacement gradually becomes difficult with the progress of soaking, because of the much closer association between the solid surface and water molecules. Curves 2K and 3K of Fig. 3 show that in the presence of the same amount of kerosene a natural floater with a larger index is more floatable than another natural floater with a smaller index. The same curves show also that the lowering of natural floatability by soaking in water is practically prevented by the presence of 1.4 mg per liter of kerosene. This suggests that the natural floatability of the suspended coal particles in flotation plants may be well preserved, because the mill water is usually contaminated by the oils used in mine and mill. A comparison between Figs. 3 and 4 reveals that a sharp decrease of natural floatability caused by soaking in water induces a small depression in actual flotation of materials with relatively small indices but results in no measurable depression of materials with large indices. This indicates that the adsorbed water molecules of natural floaters can be effectively displaced by oil droplets, particularly when the index of the natural floater is large.

The question of whether pure sulphur and graphite possess natural floatability has been a subject of much dispute.[2, 13, 14] Table III shows that pure clean sulphur and graphite exhibit measurable air-avidity in distilled water, though the organic-contaminated surface of the natural samples have higher natural floatability. The fact that previous investigators[2] failed to find a definitely measurable contact angle with pure sulphur, graphite, and bituminous coal

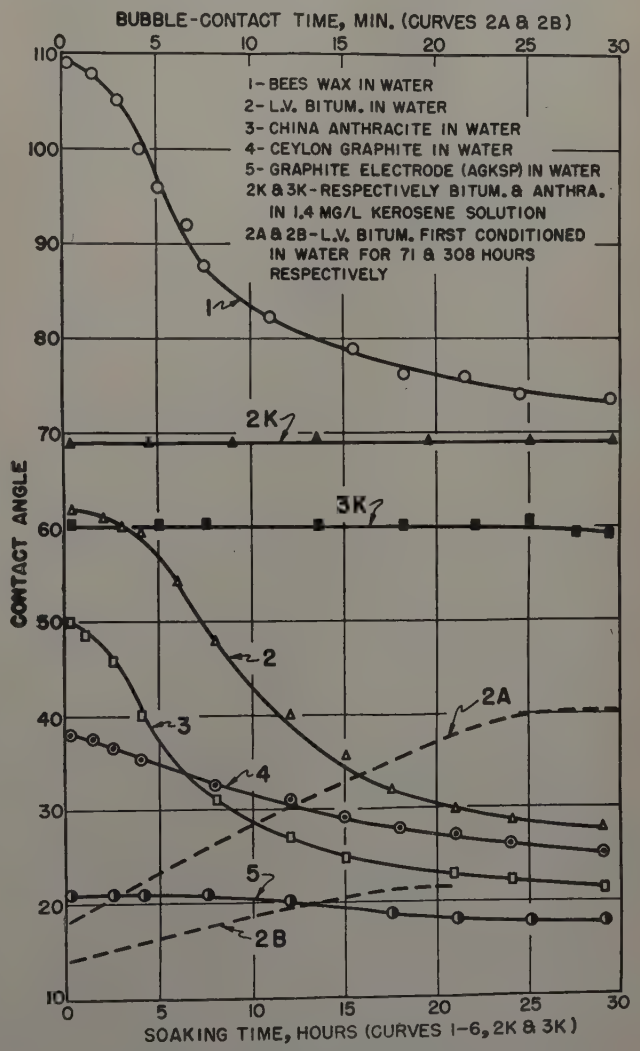


Fig. 3—Effects of soaking time and bubble-contact time on the contact angle of natural floating minerals in distilled water and kerosene solutions.

should not be interpreted as a denial of the high purity of the present samples, but rather an indication that the previous samples might be covered by a thin film of polishing powder, as suspected by Cox and Wark.[2] By adoption of the method described by Taggart and his associates,[2] the pure sulphur was prepared from a C. P. crystalline sulphur by recrystallization from thiophene-free benzene followed by nitrogen treatment. The pure graphite was made from a spectrographic electrode of AGKSP grade, furnished by the National Carbon Co., Union Carbide and Carbon Corp. The electrode was guaranteed to be 99.99+ pct pure graphitic carbon. The electrode was cut into several rectangular sticks, about 1 cm long, 0.3 cm wide, and 0.3 cm thick. These sticks were placed in the graphite tube of a graphitizing furnace,[15] and a stream of helium gas was passed constantly through the tube to prevent any reaction which would contaminate the graphite sticks or the clean graphite tube. The temperature of the tube was raised by passing a large electric current through it at reduced voltage. After the temperature reached 3100°C and remained there for 1 hr, the tube was cooled to room temperature.

Table III. The Contact Angle of Sulphurs and Graphites in Distilled Water and in Kerosene Solutions

Test No.	Material	Distilled Water		Solution Contains 1.4 Mg Per Liter Kerosene	
		Induction Time, Min	Average Contact Angle	Induction Time, Min	Average Contact Angle*
1	Roll sulphur	0.5	44	0.5	73
2	Roll sulphur	1.0	46	1.0	73
3	Precipitated sulphur	0.5	38	0.5	52
4	Precipitated sulphur	1.0	40	1.0	55
5	Purified sulphur	0.5	23	0.5	39
6	Purified sulphur	1.0	25	1.0	41
7	Ceylon graphite	0.5	28	0.5	52
8	Ceylon graphite	1.0	30	1.0	53
9	Spectrographic electrode AGKSP, Union Carbide & Carbon	1.0	21	1.0	32
10	Purified spectrographic electrode AGKSP	0.5	17	0.5	24
11	Purified spectrographic electrode AGKSP	1.0	18	1.0	25
12	Purified spectrographic electrode AGKSP	2.0	19	2.0	25
13	Graphite stick, Speer Carbon Co.	0.5	23	0.5	34
14	Graphite stick, Speer Carbon Co.	1.0	24	1.0	35
15	Nitrogenous graphite stick, Speer Carbon Co.	0.5	22	0.5	29
16	Nitrogenous graphite stick, Speer Carbon Co.				

* Contact angle was measured at the end of 2-min conditioning time.

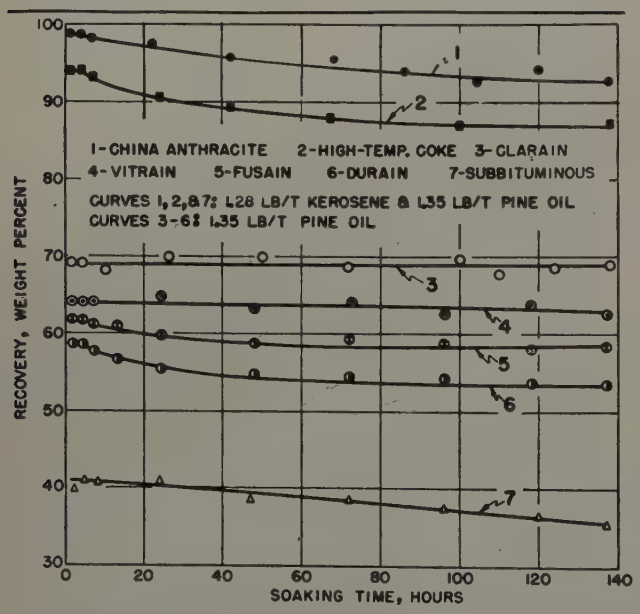

RECOVERY, WEIGHT PERCENT
SOAKING TIME, HOURS
1-CHINA ANTHRACITE 2-HIGH-TEMP. COKE 3-CLARAIN
4-VITRAIN 5-FUSAIN 6-DURAIN 7-SUBBITUMINOUS
CURVES 1,2,&7: L28 LB/T KEROSENE & L35 LB/T PINE OIL
CURVES 3-6: L35 LB/T PINE OIL

Fig. 4—Effect of soaking in distilled water on the actual flotation of various minerals.

Table IV. Relationship Among the Calculated Floatability Index, the Wettability or Hydrophobic Property, and the Adsorbability for Purified Kerosene of Minerals in Distilled Water at Room Temperature

Mineral	Approximate Amount of Purified Kerosene Adsorbed by Minerals of 48x65 Mesh, Mg Per G	Approximate Wettability of Minerals in Distilled Water		Calculated Floatability Index (Using Ceylon Graphite as Standard)
		Particle Size, Mesh	Time, Min	
Wood rosin K	56.8	10x14	25.0	127.8
Low volatile bituminous	32.6	20x28	18.0	105.9
Roll sulphur	24.8	35x48	11.0	
Ceylon graphite	19.7	48x65	10.0	100.0
Clarain (h. v. bitum.)	19.5	20x28	5.0	80.8
Vitrain (h. v. bitum.)	18.6	20x28	4.0	76.4
China anthracite	17.2	28x35	3.0	66.3
Fusain (h. v. bitum.)	12.8	28x35	0.8	60.6
Durain (h. v. bitum.)	11.4	28x35	1.7	55.7
High temp. coke C.	9.3	35x48	1.1	63.2
Subbituminous C	8.0	20x28	1.5	61.3
Semianthracite	5.1	35x48	0.3	49.7
Anthracite bone	4.7	28x35	0.1	52.8
Lignite	1.4	20x28	0.9	22.5
Oxidized h. v. bitum.[a]	1.3	28x35	0.04	
Oxidized h. t. coke	1.2	35x48	0.03	
Carbonaceous slate	1.1	48x65	0.01	
Calcite	0.8	65x100	inst.	
Pyrite	0.7	48x65	inst.	
Quartz	−0.2	48x65	inst.	

[a] Oxidized with concentrated nitric acid; inst., instantaneously wetted.

Adsorption of Oils by Minerals

The premise that the floatable components are much more oil-avid than the non-floatable components was verified by testing the adsorbability of minerals for neutral oils. Table IV shows that the adsorbability of the tested minerals for purified kerosene generally increases with an increase of the calculated floatability index of the mineral. This is also true for the relationship between adsorbability and hydrophobic property, because the hydrophobic property of a mineral is roughly proportional to its calculated index. Table IV shows also that the adsorbability of coal and coke for kerosene is sharply lowered by oxidation,[8] owing to the increase of oxy-

Table V. The Adsorbability of 48x65 Mesh Ceylon Graphite for Various Liquid Hydrocarbons in Distilled Water at Room Temperature

Hydrocarbon		Amount of Hydrocarbon Adsorbed by 1 G of Graphite, Mg Per G	Number of Hydrocarbon Molecules Adsorbed by 1 G of Graphite
Name	Solubility G/1		
Benzene	0.82^{20}	0.037	2.86×10^{17}
Cyclohexane	i	0.093	6.69×10^{17}
Cyclohexene	v. sl. s.	0.462	3.41×10^{18}
Hexane	0.183^{15}	0.277	1.95×10^{18}
1-Hexene	i	0.343	2.47×10^{18}
Decane	i	0.774	3.29×10^{18}
Undecane	i	1.280	4.96×10^{18}
Dodecane	i	6.230	2.22×10^{19}
Tridecane	i	8.091	2.66×10^{19}
Tetradecane	i	67.425	2.08×10^{20}
Hexadecane	i	91.930	2.46×10^{20}
O-thiocresol	i	50.338	2.45×10^{20}
Gasoline	sl. s.	0.470	—
Kerosene	i	16.680	—

i, insoluble; v. sl. s., very slightly soluble; sl. s., slightly soluble.

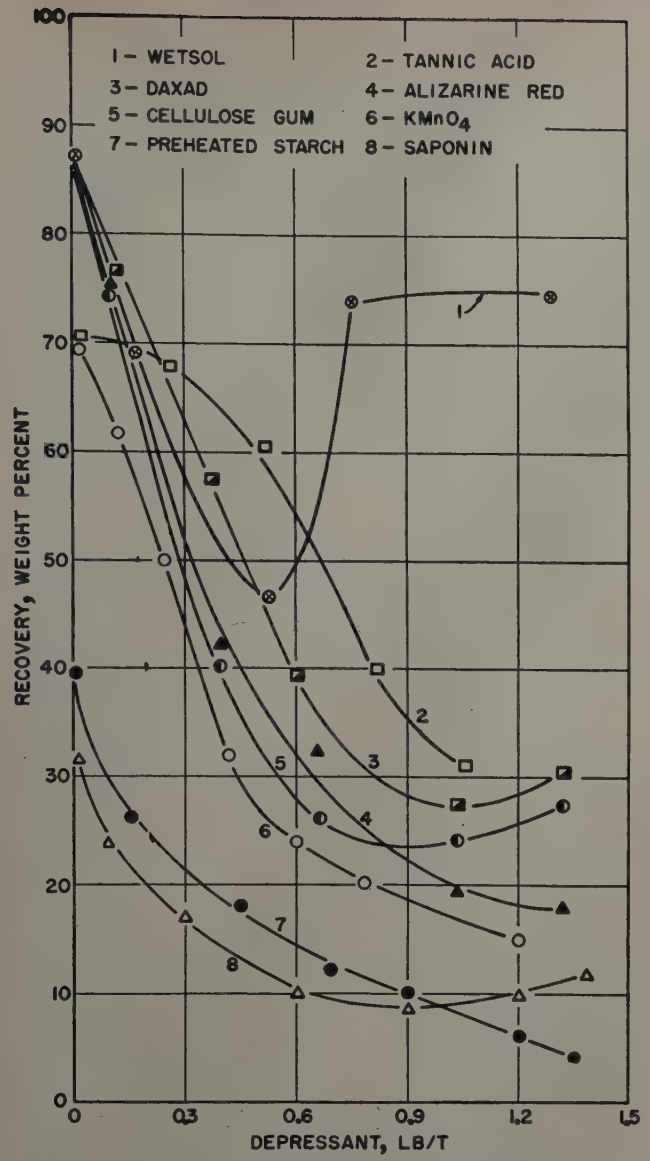


Fig. 5—Effect of various depressants on the flotation of a bituminous coal filter cake from Nemacolin, Pa. Curves 1, 3 to 5: 0.18 lb per ton Pine Oil. Curves 2 and 6: 0.14 lb per ton Pine Oil. Curve 7: 0.11 lb per ton Pine Oil. Curve 8: 0.09 lb per ton Pine Oil.

gen contents and consequently hydrophilic properties. Fig. 5 indicates that the lowering of adsorbability for oil occurs also when the surface of coal is coated with hydrophilic colloids. Table V shows that the amount of liquid paraffin hydrocarbons adsorbed by Ceylon graphite generally increases with an increase of the number of carbon atoms of the hydrocarbon. This may be caused by the fact that the hydrophobic property of these hydrocarbons, as partly indicated by solubility, gradually increases in going up the homologous series. Generally speaking, the adsorbability of minerals for neutral oils is facilitated by an increase in the hydrophobic property of the mineral and/or the neutral oil, and is inhibited or at least lowered whenever the surface of the mineral is rendered completely hydrophilic.

The above experimental data led to the postulate that oiling is a phenomenon of *hydrophobic adsorption,* because it results from mutual attraction between the hydrophobic molecules of neutral oils and the hydrophobic or floatable components of a solid surface. According to the established treatise[16] on the work of adhesion and adhesion tensions of liquids against solids, the adhesive force between an oil and a hydrophobic solid is much larger than that between the oil and a hydrophilic solid, or between water and hydrophobic solid. Although the interfacial energy of solid-liquid is not determinable by direct experimentation, the related contact angle is. The Van der Waals forces,[17] having the peculiar property of bringing like atoms together, are also expected to appear in the hydrophobic adsorption when the surface atoms of the oil and the mineral are alike. Furthermore, forces of a chemical and an ionic character cannot be ruled out entirely[18] when the oil and mineral used are not totally free of residual chemical valences, partial dipoles,[19] or polarizability.[20] Under suitable conditions, the forces involved in the adsorption of an oil by a solid are sufficient to overcome cohesive forces within the oil and thus affect a monolayer spreading of the oil over the solid surface. Yet the presence of an excessive amount of oil can create a multilayer of oiling.

An attempt was also made to ascertain the effect of crystal structure on oiling. Fig. 6 shows schematically that the molecules of alkyl hydrocarbons and the aromatic nuclei of aryl hydrocarbons can be superimposed on the molecules of solid hydrocarbons, graphite, coal, sulphur, and the sulphur layer of molybdenite. This is in line with the speculation of Van der Waarden[21] that oil molecules are adsorbed flatly upon the surface of carbon black. It would be highly tempting to regard the oiling of these solids to be largely caused by the geometrical compatibility of lattice dimensions, were it not that a superposition of oils on cellulose is also possible and that as yet no oiling seems to occur on this material. Compared with the important role played by the kind of surface components or the magnitude of hydrophobic property of the mineral and oil used, the influence exerted by the geometrical compatibil-

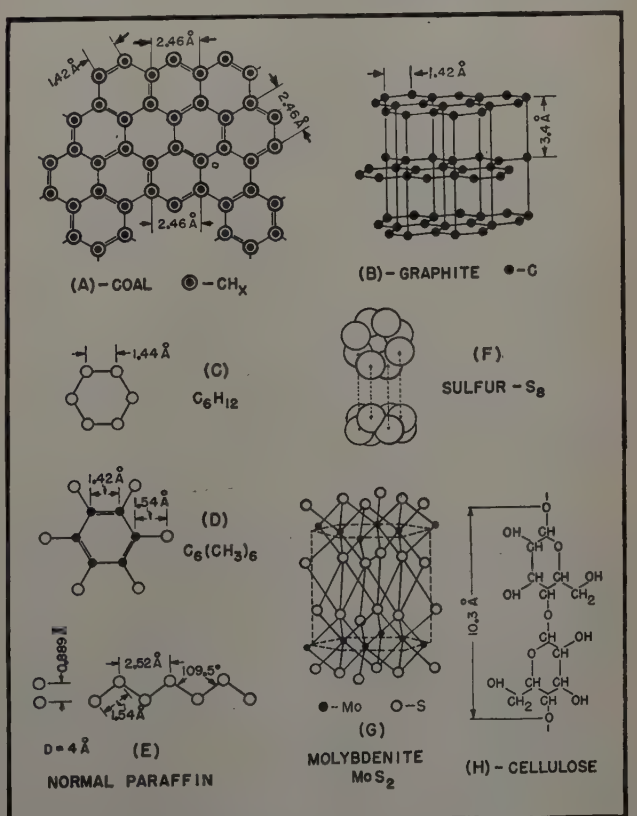


Fig. 6—The molecular structure of coal, graphite, hydrocarbons, sulphur, molybdenite, and cellulose.

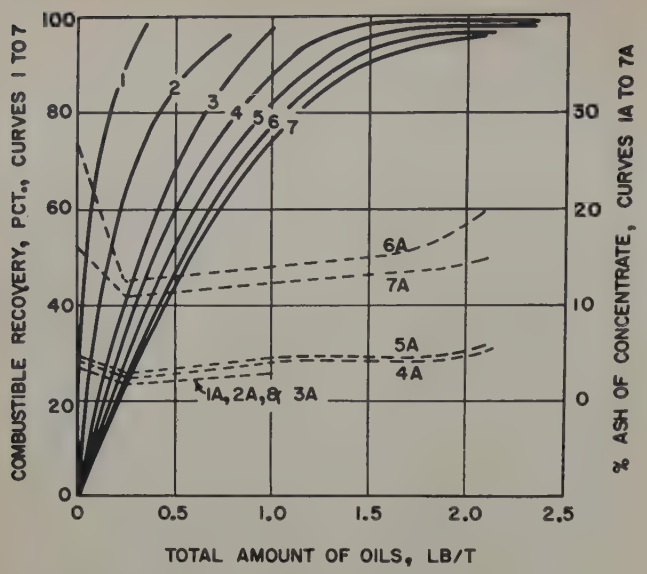


Fig. 7—Flotation of different petrographic components taken from various bituminous coals. Curves 1 and 2 are, respectively, vitrain and fusain obtained from a medium volatile bituminous coal, Springdale, Pa. Curve 3 is vitrain obtained from a high volatile—A bituminous coal, Pikeville, Ky. Curves 4 to 7 are, respectively, clarain, vitrain, fusain, and durain obtained from a high volatile-B bituminous coal, LaSalle County, Illinois.

ity between the substratum and the superstratum is of secondary importance.

The application of the surface components hypothesis in explaining the floatability of the petrographic components[6d] of coals was experimentally verified. Curves 4 to 7 of Fig. 7 and tests 26 to 29 of Table I show that the floatability of petrographic components taken from a high volatile-B bituminous coal decreases in the order of clarain, vitrain, fusain, and durain, corresponding to the same order of their calculated floatability indices. This phenomenon also holds true for the petrographic components of a medium volatile bituminous coal, as shown in curves 1 and 2 of Fig. 7 and tests 18 and 19 of Table I. Furthermore, Fig. 7 and Table I show that the petrographic components of a highly floatable coal are more amenable to flotation than the corresponding petrographic components of a moderately floatable coal. For example, curves 1, 3, and 5 of Fig. 7 show that the vitrain of a medium volatile bituminous coal is more floatable than the vitrain of a high volatile-A bituminous coal, while the latter is more floatable than the vitrain of a high volatile-B bituminous coal. Table I shows that the calculated floatability index of the parent coals and that of the vitrains decrease in the same order. This is also true for fusain, as shown in curves 2 and 6 of Fig. 7.

Fig. 8 shows that the floatability of coals, carbons, and hydrocarbon minerals belonging to the same rank can be roughly correlated with their contents of fixed carbon, volatile matter, and moisture. The ash-floatability curves, not presented, showed no significant correlation. These phenomena should not be interpreted as a denial but rather as a further confirmation of the surface components hypothesis. For example, curves 1 to 6 of diagram *A* show that the floatability of coals and carbons of the same rank is directly proportional to their fixed carbon contents. This is partly attributed to the fact that the difference in hydrocarbon content between materials of the same rank is relatively small, and partly to the fact that the material of higher carbon content usually has less moisture and oxygen than another material of lower carbon content, as indicated in Table I. Conversely, curve 7 of diagram *A* shows that the floatability of hydrocarbon minerals generally decreases with the increase of fixed carbon content. This can be explained by the fact that a high content of remnant carbon atoms of these minerals is usually accompanied by a low content of hydrocarbon molecules, as revealed by actual calculations. It is rather difficult to compensate for a lowering of hydrocarbon content by increasing the carbon content, because the former is much more floatable than the latter. The carbon content of all materials, except that of lignitic coals, used in diagram *A* of Fig. 8 were calculated to dry and ash-free basis. A moist and ash-free basis was found to be more suitable for lignite.

Diagram *B* of Fig. 8 brings out two significant points. First, curves 1 to 4 show that, within the same rank, floatability of carbons and coals of subbituminous rank or higher increases as the volatile matter content decreases. This is largely because the volatile matter of these materials, being poor in hydrocarbon compounds and rich in moisture and carbon dioxide, is relatively low in floatability, and partly because, within the same rank, a high volatile coal usually contains less carbon and more moisture and oxygen than a low volatile coal, while the difference in hydrocarbon content between these two coals is relatively small. Second, in contrast to the above relationship, curves 5 and 6 of diagram B show that the floatability of hydrocarbon minerals and lignitic coals, within the same rank, generally increases as the volatile matter content increases. This is chiefly caused by the fact that the volatile matter of these materials, being rich in hydrocarbon compounds and poor in moisture and carbon dioxide, is highly floatable.

Diagram *C* of Fig. 8 shows that the floatability of

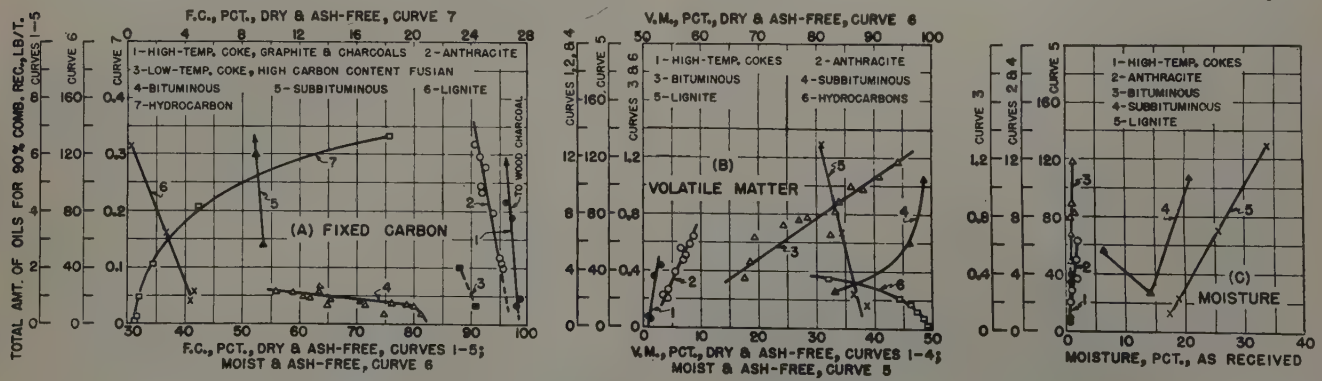


Fig. 8—Correlation between proximate components and floatability of coals, carbons, and hydrocarbon minerals.

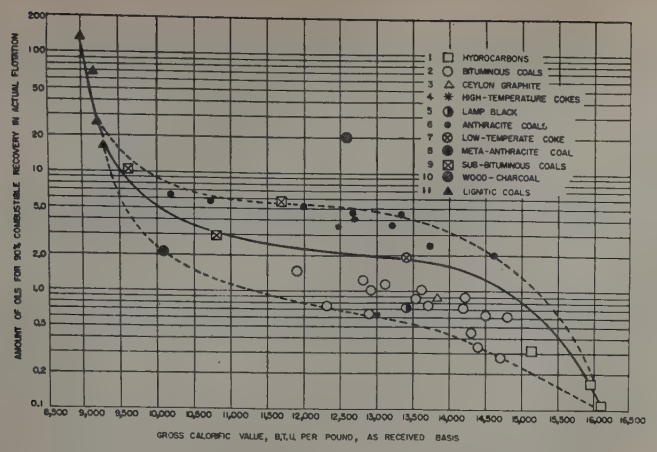


Fig. 9—Relation between the gross calorific value and the floatability of coals, cokes, and hydrocarbon minerals.

materials belonging to the same rank generally decreases with the increase of their moisture contents. The lack of any significant ash-floatability relationship is due to the fact that a large portion of the gangue minerals was liberated from coals, carbons, and hydrocarbon minerals by reduction to the —48 mesh flotation size. Thus the result of ash analysis of the flotation feed is not indicative of the amount of the interlocked gangue minerals and consequently has very little bearing on the floatability of materials tested.

Fig. 9 shows that there is a general tendency for coals, carbons, and hydrocarbon minerals of higher calorific values to have higher floatabilities. This is because the heat of combustion[22,23] of the floatable components is much larger than that of the nonfloatable components. The lack of a smooth floatability-calorific value relationship may find its explanation in the fact that the floatability and the heat of combustion of the chemical components of the tested materials are not exactly in direct proportion. This can be visualized by comparing Eq. 2 of this paper and Dulong's formula,[6e] widely used for calculating the calorific value of coal.

Summary

1—A surface components hypothesis is proposed to explain the different floatabilities of coals, carbons, and hydrocarbon minerals, floated with a neutral oil and a frother or with a frother only.

2—The actual floatability, natural floatability, and adsorbability for neutral oils of coals, carbons, and hydrocarbon minerals generally increase with the increase of their calculated floatability indices.

3—The calculated index required for a material to exhibit a clinging contact angle in distilled water alone is around 65.

4—Oiling is facilitated by an increase in the hydrophobic property of the mineral and/or the neutral oil and is inhibited whenever the surface of the mineral becomes completely hydrophilic.

5—The floatability of petrographic components not only is directly proportional to the floatability of their parent coals, but also decreases in the order of clarain, vitrain, fusain, and durain, corresponding to the same order of their calculated indices.

6—The floatability of coals, carbons, and hydrocarbon minerals belonging to the same rank can be roughly correlated with their contents of fixed carbon, volatile matter, and moisture.

Acknowledgment

The writer wishes to thank Messrs. H. T. Darby, C. D. Neubling, and S. R. Milliken for their kind assistance in chemical analysis. He is indebted to Dr. M. G. Boobar and Dr. P. L. Walker, Jr. for the purification and nitrogenation of the graphite samples. Thanks are also extended to Messrs. R. Kuchinic and G. Simkovich for proof-reading the manuscript, and to Professor D. R. Mitchell and Dr. H. B. Charmbury for their active interest in the prosecution of this work.

References

[1] E. T. Wilkins: Coal Preparation: Some Development to Pulverize Fuel Practice. Conference on Pulverized Fuel, Harrogate, England (1947) p. 398.

[2] A. F. Taggart, G. R. M.Del Giudice, A. M. Sadler, and M. D. Hassialis: Oil-Air Separation of Nonsulphide and Nonmetal Minerals. *Trans.* AIME (1939) **134**, p. 180.

[3] A. M. Gaudin: *Principles of Mineral Dressing,* p. 186. McGraw-Hill Book Co., New York, 1939.

[4] C. Golumbic, J. B. Anderson, M. Orchin, and H. H. Storch: Solvent Extraction of Coal by Aromatic Compounds at Atmosphere Pressure, U. S. Bur. Mines *R.I.* 4662 (March 1950).

[5] R. L. Hubbard and K. E. Stanfield: Laboratory Study of Asphalts from Native Bitumens and Bituminous Sandstone. U. S. Bur. Mines *R.I.* 4523 (August 1949).

[6] H. H. Lowry: *Chemistry of Coal Utilization.* a, p. 677; b, p. 425; c, p. 450; d, p. 86; e, p. 132. John Wiley & Sons, New York, 1945.

[7] A. H. Warth: *The Chemistry and Technology of Waxes,* p. 212. Reinhold Publishing Corp., New York, 1947.

[8] S. C. Sun: Effects of Oxidation of Coals on Their Flotation Properties. Paper presented at AIME Annual Meeting, St. Louis, Mo. (February 1951).

[9] H. B. Charmbury, J. W. Eckerd, J. S. LaTorre, and C. R. Kinney: The Chemistry of Nitrogen in Humic Acids from Nitric Acid Treated Coals. *Journal of American Chemical Society* (1945) **67**, p. 625.

[10] P, L. Walker, Jr.: Some Factors Affecting the Mechanism of the Carbon-Carbon Dioxide Reaction, Ph.D. thesis, Pennsylvania State College (1952) p. 13-16.

[11] A. M. Gaudin and S. C. Sun: Correlation Between Mineral Behavior in Cataphoretic Cell and in Flotation. *Trans.* AIME (1946) **169**, pp. 347.

[12] H. E. Blayden, J. Gibson, and H. L. Riley: An X-Ray Study of the Structure of Coals, Cokes, and Chars, *Proc.* Conference on the Ultra-Fine Structure of Coals and Cokes, London, (1944) p. 176.

[13] H. L. Sulman: A Contribution to the Study of Flotation, *Trans.* Institution of Mining and Metallurgy, London, (1919) **29**, p. 44.

[14] A. M. Gaudin: *Flotation,* pp. 38, 342. McGraw-Hill Book Co., New York, 1932.

[15] J. J. Zelinski: A Study of the Surface Oxides of Graphite at Elevated Temperatures, Ph.D. thesis, Pennsylvania State College, (1950) pp. 48 and 90.

[16] N. K. Adam: *The Physics and Chemistry of Surfaces,* 3rd ed. pp. 173-208. Oxford University Press, London, 1941.

[17] A. E. Van Arkel: *Molecules and Crystals in Inorganic Chemistry,* p. 174. Interscience Publishers Inc., New York, 1951.

[18] M. Van der Waarden: Stabilization of Carbon-Black Dispersions in Hydrocarbons. *Journal of Colloid Science* (1950) **5**, p. 317.

[19] A. E. Van Arkel: Mutual Solubility of Liquids, *Trans.* Faraday Society, (1946) **42B**, p. 81.

[20] L. Pauling: *The Nature of the Chemical Bond,* p. 152. Cornell University Press, Ithaca, N. Y., 1945.

[21] M. Van der Waarden: Adsorption of Aromatic Hydrocarbons in Nonaromatic Media on Carbon Black. *Journal of Colloid Science* (October 1951) **6**, p. 443.

[22] J. Griswold: *Fuels, Combustion and Furnaces,* p. 130. McGraw-Hill Book Co., New York, 1946.

[23] E. Klepetko: Recovery of Resin From Utah Coal, *Trans.* AIME (1948) **177**, p. 433.

The Federal Coal Mine Safety Act

by J. J. Forbes

THE Federal Coal Mine Safety Act (Public Law 552, 82nd Congress) was approved on July 16, 1952. It incorporates, as Title I, the Coal Mine Inspection and Investigation Act of May 7, 1941 (Public Law 49, 77th Congress), which gave Federal inspectors only the right to enter coal mines for inspection and investigation purposes but no power to require compliance with their recommendations. Title II contains the enforcement provisions of the act; its purpose is to prevent major disasters in coal mines from explosions, fires, inundations, and man-trip or man-hoist accidents.

At this point a brief account of events that preceded the enactment of the Federal Coal Mine Safety Act seems appropriate. The hazardous nature of coal mining was recognized by the Federal Government as long ago as 1865, when a bill to create a Federal Mining Bureau was introduced in Congress. Little was done, however, until a series of appalling coal-mine disasters during the first decade of this century provoked a demand for Federal action. As a result an act of Congress established a Bureau of Mines in the Department of the Interior on July 1, 1910. The act made it clear that one of the foremost activities of the Bureau should be to improve health and safety in the mineral industries. One of the first projects selected by the small force of engineers and technicians then employed was to determine the causes of coal-mine explosions and the means to prevent them. By investigations after mine disasters the fundamental causes and means of prevention were soon discovered, and the coal mining industry was informed accordingly. However, despite this knowledge and the enactment of State laws and the Federal Coal Mine Inspection and Investigation Act of 1941, mine disasters continued to occur with disheartening frequency and staggering loss of life. The devastating explosion at the Orient No. 2 mine on December 21, 1951, resulted in the death of 119 men. The Orient disaster rekindled the memory of the Centralia, Ill., disaster of March 25, 1947, which caused the death of 111 coal miners. These two tragedies ultimately brought about enactment of the Federal Coal Mine Safety Act.

The act is a compromise measure. Senator Matthew M. Neely of West Virginia and Congressman Melvin Price of Illinois introduced almost identical versions in the 82nd Congress, but they were considered too drastic. The final version was introduced by Congressman Samuel K. McConnel, Jr., of Pennsylvania, after considerable discussion and amendment in committee hearings. It was passed by the Congress and became effective when signed by the President on July 16, 1952.

The act is somewhat limited in scope because it applies only to approximately 2000 coal mines in the United States and Alaska that employ regularly 15 or more individuals underground. It exempts approximately 5300 mines employing regularly fewer than 15 individuals underground and all strip mines, of which there are about 800. Moreover, it covers only conditions and practices that may lead to major disasters from explosion, fire, inundation, or man-trip or man-hoist accidents. According to Bureau records, such accidents have resulted in less than 10 pct of all the fatalities in coal mines. It is important to mention that the law is not designed to prevent the day-to-day type of accidents that have caused the remaining 90 pct or more of the fatalities, because it was the specific intention of the Congress to reserve the hazards which caused them to the jurisdiction of the coal-producing states.

Many who opposed any Federal legislation that would give the Federal inspectors authority to require compliance with mine safety regulations claimed that such legislation would usurp or infringe upon States' rights. To assure that the principle of States' rights would be preserved, the act provides for joint Federal-State inspections when a state desires to cooperate in such activities. The Director of the Bureau of Mines is required by the act to cooperate with the official mine-inspection or safety agencies of the coal-producing states. The act provides further that any state desiring to cooperate in making joint inspections may submit a State plan for carrying out the purposes of this part of the act. Certain requirements are listed; these must be met by a state before the plan can be accepted. The Director of the Bureau of Mines, however, is required to approve any State plan which complies with the specified provisions. The Director may withdraw his approval and declare such a plan inoperative if he finds that the State agency is not complying with the spirit and intent of any provision of the State plan.

When this paper was prepared, agreements for joint Federal-State inspections had been entered into with Wyoming and Washington. A few other states have indicated their desire to submit a State plan and negotiations toward that end are now under way. Reluctance to enter into such agreements may be due to the mine operators' knowledge that in the states that adopt a cooperative plan they are prohibited from applying to the Director of the Bureau of Mines for annulment or revision of an order issued by a Federal inspector and must appeal directly to the Federal Coal Mine Safety Board of Review for such action. Experience has proved that review by the Director as provided in the act is a less expensive and time-consuming procedure to all concerned than applying to the Board. Reluctance also may stem from the fact that joint Federal-State inspections somewhat restrict the movements of the State mine inspectors and tend to reduce the number of inspections of mines.

Where a State plan is not adopted, the Federal coal mine inspector is responsible under the law to take one of two courses of action if he finds certain hazardous conditions during his inspections.

The first action involves imminent danger. If a Federal inspector finds danger that a mine explosion, mine fire, mine inundation, or man-trip or man-hoist accident will occur in a mine immediately or before the imminence of such danger can be elim-

J. J. FORBES, Member AIME, is Director, Bureau of Mines, U.S. Department of Interior, Washington, D. C.

Discussion on this paper, TP 3678F, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, Feb. 18, 1953. Los Angeles Meeting, February 1953.

inated, he issues a written order requiring the operator of the mine to cause all persons, except those specifically permitted by law to correct the conditions, to be withdrawn from and debarred from entering the area described in the order. The operator is not permitted under the act to resume normal operations in such an area until 1—the conditions specified in the order are corrected to the satisfaction of three duly authorized representatives of the Bureau of Mines appointed by the Director after receipt of an application from the operator for annulment or revision of such order, or 2—the Board of Review finds that after receipt of an application from the operator for annulment or revision of the order and after a formal hearing, the condition described was erroneous or no longer exists.

The second action involves one or more violations of the mine-safety provisions set forth in the act. If a Federal inspector finds that any mine-safety provision is being violated and that the violation does not cause danger that an explosion, fire, inundation, or man-trip or man-hoist accident will occur in such mine immediately or before the imminence of such danger can be eliminated, he is required to issue a written notice to the operator, giving the operator a reasonable time for correcting the violation. If upon making his check inspection to determine whether the violation has been abated in the time originally specified, the Federal inspector finds that the violation has been totally abated, he issues another written notice to that effect. If the inspector finds that the operator should be given additional time to abate the violation, he grants an extension in writing. If, however, the violation is not abated and the Federal inspector finds that circumstances, such as obvious lack of effort by the operator to correct the condition, do not justify granting an extension of time, he issues a written order requiring the operator to cause all persons, except those specifically permitted by law to correct the conditions, to be withdrawn from and debarred from entering the area described in the order. Here again the operator is prohibited from resuming normal operations in the affected area until the order is annulled or revised by the Director of the Bureau of Mines or by the Board of Review under the procedure described for imminent dangers.

It is appropriate to point out that formal applications to the Director for annulment or revision of actions taken by a Federal inspector or applications to the Board of Review must be based on orders issued by the inspectors. Neither the Director nor the Board can consider any formal action on an application to review any action of an inspector other than an order. If, however, an operator feels that he has been unjustly cited under the act for violating any mine-safety provision and if, after consultation with the inspector and the Bureau's appropriate field officials, the issues cannot be resolved, such an operator is free to present his case to the Director. Every effort will be made to resolve the differences fairly and impartially.

The act provides, of course, for judicial review by the United States Court of Appeals, and eventually the United States Supreme Court, of any final order that is issued by the Federal Coal Mine Safety Board of Review.

As has been stated, the mine-safety provisions of the act cover only those dangers inherent in coal mining that cause a relatively small percentage of the total accidents. It was clearly the intention of the Congress to cover only hazards that lead to major disasters and not to enter the field of ordinary hazards. But if the safety provisions specified in the act are complied with conscientiously by all concerned, particularly when a Federal inspector is not on the premises, major disasters will be rare.

The law provides penalties only for failure to comply with any order issued by a duly authorized representative of the Bureau of Mines. The penalties, of course, will be invoked only when all other measures to gain compliance fail. It should be pointed out, however, that the law contains no penalties for non-compliance with the mine-safety provisions between inspections of the Bureau's authorized representatives.

The act recognizes State mining laws by providing that any State or Territorial law, in effect before or after the effective date of the Federal Coal Mine Safety Act, which provides greater safety than the latter on the same phases of mining shall not be construed to conflict with the act.

When this paper was prepared the Federal Coal Mine Safety Act had been in effect for six months. Much has been accomplished during that short period. According to the records, 8007 coal mines were operated during 1952. Of these 1919 were Title II mines employing regularly 15 or more persons underground. The remainder were Title I mines, including 5281 deep mines employing regularly fewer than 15 men underground, as well as 807 strip mines.

During the six-month period after the act became effective, Bureau of Mines inspectors examined 1352 Title II mines in the United States and Alaska. They found 232, or 17 pct, free of violations of the act during their inspections. In all, 3914 violations of the mine-safety provisions were observed in the remaining 1120 mines; 1738 violations, or 44 pct, were corrected in 268 mines before the inspections were completed. Notices were not issued in instances wherein violations of the mine-safety provisions were totally abated, as determined on the spot by the inspector after inspection of the mine was completed. Except for the few instances that required issuance of withdrawal orders because of imminent danger, the operators of the other 852 mines were granted reasonable time to abate the 2176 violations, because these could not be corrected immediately. Bureau of Mines records show that about two-thirds of all the violations of the mine-safety provisions for which written notices were issued were corrected in the time originally specified. Except in a few instances requiring withdrawal orders, the inspectors granted extensions of time to correct the remaining third of the violations.

At three mines the Federal inspectors issued four withdrawal orders because of imminent danger. The imminent dangers cited were: 1—inundation, 2—likelihood of a man-trip accident owing to bad roof over haulageway (two mines), and 3—explosion. Thirteen withdrawal orders have been issued at seven mines because the violations were not abated in the time originally specified, and the inspectors decided that extensions of time were unwarranted. Twenty-one mines have been classed as gassy according to the act, primarily because the inspectors collected therein samples of mine atmospheres containing 0.25 pct or more of methane since the effective date of the act. This last statement should not be construed to mean that only 21 of the 1352 Title II mines, inspected during the first six months of operating under the act, were gassy mines. If a mine

is classed gassy by the State, before or after the effective date of the act, the operator of such mine must comply with all provisions of the Federal Coal Mine Safety Act pertaining to gassy mines; therefore, those mines that are classed gassy by State law are also classed gassy by the Federal act.

It is interesting to note that as yet not one withdrawal order has been appealed to the Federal Coal Mine Safety Board of Review. In each instance where a withdrawal order has been issued and the operator appealed to the Director of the Bureau of Mines claiming that the dangerous condition was corrected, such appeals have been acted upon promptly, regardless of whether they were received on holidays, Saturdays, Sundays, or regular working days. The Bureau is as much interested as the operators in causing no undue delay in reopening mines after violations of the law are corrected. In each instance, after prompt special investigation by three duly authorized representatives of the Bureau of Mines, the Director verified that the conditions had been corrected, the inspector's order was annulled forthwith, and the mine was permitted to resume normal operation.

Two of the 21 orders classifying mines as gassy were appealed to the Director. One appeal was denied by the Director and was carried to the Board of Review; the Board sustained the Director's decision. The other appeal was denied by the Director and was pending with the Board when this paper was completed.

The success of the Federal Coal Mine Safety Act cannot be measured by the number of violations discovered by Federal inspectors, since the act requires only one annual Federal inspection of each mine coming within its purview. The important factor is not what the mine operator does for safety when a Federal inspector is on the premises but what is done during the long periods when a Federal inspector is not there. This will be reflected in the injury rates for the mine and the entire coal-mining industry. The following information should be interesting. The calendar year 1952 was the best in the history of the American coal-mining industry with respect to the number of fatalities and the fatality rate based on million man-hours of exposure. The total number of men killed in 1952 was 546, and the fatality rate was 0.84. It is undeniable that these are 546 too many lives lost. When viewed in perspective, however, it represents considerable progress in accident prevention over the years. The figure compares favorably with 1471 coal-mine fatalities in 1942, the first full year of operation under the Coal Mine Inspection and Investigation Act of 1941, and the average of 2500 fatalities annually when the Bureau of Mines was established in 1910. Non-fatal injury rates have not improved in percentage as well as the fatal rates, but reporting of non-fatal injuries in former years was not nearly as complete as in recent years.

Present indications are that the act has increased the safety consciousness of both officials and mine workers alike. To achieve the ultimate in mine safety, all concerned must believe in it and work diligently for it. The stimulating force of a safety program must begin with top management, but to be successful it must not end there. If everyone concerned will accept his responsibility and comply with the spirit and intent of the act, the reward will be in helping to avoid the terrible waste of human values and in helping to place the coal-mining industry in a favorable safety position with respect to other important industries.

Commercial Synthesis of Star Sapphires And Star Rubies

by Clifford Frondel

THE aluminum oxide known as corundum has several varieties that have been used as gem materials since ancient times. These include the red variety called ruby, the blue variety sapphire, and the asteriated types of ruby and sapphire. Asteriated sapphire was known to the ancients under the name of *astrios*, and descriptions of this gem were given by Pliny and other early writers. The relation of these gem materials to each other as varieties of a single species, corundum, was first clearly established by Haüy in 1805 and J. M. Güthe[1] in 1809. Natural gem corundum is obtained principally in Ceylon, Siam, Burma, India, and Australia.

Star sapphires and rubies always have been highly prized and in recent years have become particularly popular in the United States. Natural star rubies of large size and fine quality are extremely rare and rank with large and fine emeralds and diamonds as the most valuable of gem stones. Interest thus attaches to the recent success in commercially synthesizing star sapphires and rubies of a size and perfection unmatched by nature.

Corundum was first synthesized as crystals in 1837 by A. Gaudin, who fused alum and aluminum oxide with potassium sulphate. Numerous other syntheses have been reported in more recent years. Summaries of this work are given in Doelter's *Handbuch der Mineralchemie,* Mellor's *Comprehensive Treatise of Inorganic Chemistry,* and other reference works. The methods employed include the reaction in the gaseous state at high temperatures of halogen compounds of aluminum with water vapor or boric oxide; the crystallization of aluminum oxide from fusion in potassium molybdate, potassium dichromate or borax; and crystallization from highly aluminous silicate melts. Red, blue, or green colored varieties can be obtained by the addition of small

C. FRONDEL is Associate Professor of Mineralogy and Curator of the Mineralogical Museum, Harvard University.

Discussion on this paper, TP 3682H, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, May 4, 1953. Los Angeles Meeting, February 1953.

amounts of chromium, iron, or other inorganic pigmenting agents. The effective pigmenting ions in general are trivalent and comparable in ionic radius to aluminum, so that they can enter into solid solution in the crystals in substitution for aluminum. It should be emphasized that these methods of synthesis do not afford single-crystals of corundum of sufficient size or clarity to be used as gem material. In these methods it is very difficult or impossible to control the inception of crystallization, so that one or only a few crystal nuclei are formed that will then grow into relatively large individuals. It is the ease of such control that is one of the main advantages of the so-called Verneuil process of synthesizing corundum.

The first synthetic corundum of gem quality was produced commercially in about 1908 by a method developed in 1900 by a French chemist, A. Verneuil. With this method finely divided aluminum oxide is fed through an oxyhydrogen flame with a temperature of about 2000°C. The aluminum oxide usually is prepared by the thermal decomposition of carefully purified ammonium alum. The droplets of molten aluminum oxide formed in the flame are allowed to fall upon a support of fire clay or of crystalline corundum maintained at a relatively low temperature. Crystallization of the aluminum oxide then ensues and a single-crystal or an aggregate of single-crystals of corundum builds up on the support. These are referred to in the trade as boules. If a fire-clay support is employed a number of crystal nuclei may form simultaneously, giving rise to a polycrystalline boule. This undesirable result can be obviated by using a short single-crystal piece or rod of corundum as the support. Any desired orientation of the crystallographic c-axis of the corundum to the direction of elongation and growth of the boule can be effected by controlling the orientation of the rod support. The process of boule growth is a continuous one, and single-crystals of corundum weighing as much as several hundred grams may be grown. Views of boule furnaces are shown in Figs. 1 and 2. The growth of boules of suitable size and quality by this method requires careful control of the experimental conditions. Details of the Verneuil method were closely guarded by the manufacturers for many years, but adequate general accounts can be obtained in the patent literature[2] and in reference works on gem stones such as those of Bauer, *Die Edelsteinkkunde*, and Michel, *Die Künstlichen Edelsteine*.

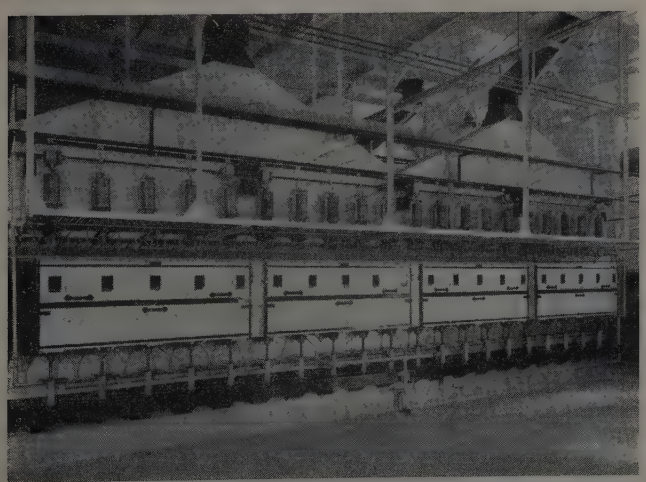


Fig. 1—Bank of boule furnaces in the Chicago plant of the Linde Air Products Co.

Fig. 2—Removing corundum boules from furnaces.

A large industry based on the Verneuil process grew up in Switzerland, France, and Germany. This has supplied yearly several thousands of pounds of synthetic corundum for use both as gem stones and as bearing materials in watches and other instruments. Since the industry was located entirely in Europe, critical shortages of synthetic corundum for technical purposes developed in the United States during World War I, and another shortage loomed at the beginning of World War II. This situation was met in 1940 by the construction of a synthetic corundum plant in the United States by the Linde Air Products Co., a subsidiary of Union Carbide and Carbon Corp. The annual U. S. production for both gem and technical purposes has been as high as 100 million carats* during the war years, but the present-day production is much less. The Verneuil process also is being used for the synthesis of single-crystals of a number of other materials valuable both for gem use and for technical purposes. These include a number of substances of the spinel-type, including gem spinel itself, $MgAl_2O_4$, together with cadmium tungstate, calcium tungstate, and ferrites, as well as rutile and other refractory oxides. Both the National Lead Co. and the Linde Air Products Co. are active in this field.

* A carat is 0.2 g.

The basic problem involved in the synthesis of star sapphires and rubies is the formation of an oriented network of acicular inclusions within the ordinary corundum boules as grown by the Verneuil method. A possible mechanism for accomplishing this is suggested by the observations of metallurgists and mineralogists on solid solutions between metals or inorganic solids. It has long been known that an originally homogeneous solid solution may become unstable and precipitate out a dissolved component, which is dispersed after precipitation in oriented fashion within the host crystal. The usual cause of the precipitation is decrease in temperature, which acts to reduce the solubility of the dissolved component, but polymorphic inversion of the host crystal, order-disorder transitions and the chemical alteration of the host crystal also are of effect in some instances.

In the case of corundum, it was found that small amounts of titanium could be introduced into solid solution in the boules. The titanium substitutes in the trivalent state for aluminum ions in the crystal structure of the corundum. If the boules are cooled

at the ordinary rate in the boule furnace in which they are grown, or are quenched in air, the titanium stays in solid solution and the boule remains transparent and homogeneous at room temperature. The solid solution is then unstable, but precipitation does not occur because of the extremely high internal viscosity of the crystal, which prevents the titanium ions from congregating and forming a separate phase. If, however, the corundum is annealed between 1100° and 1500°C in an oxidizing atmosphere for a period of time ranging upwards from a few hours, the increased thermal mobility of the titanium ions permits them to sort out. The composition of the solid solution then adjusts itself to the limiting solubility of the titanium in the corundum at the annealing temperature by the precipitation of needle-like crystals of titanium dioxide, rutile. The precipitation involves an oxidation of the titanium from the trivalent to the quadrivalent state with a concomitant diffusion of oxygen into the crystal structure. The rate of precipitation decreases with decreasing temperature and below about 1100°C is effectively nil. Precise control of the annealing conditions is important, to control the crystal size of the rutile. Over about 1500°C precipitation does not occur, and star corundum containing precipitated rutile can be homogenized by solution of the rutile by heating over about 1500°C. The titanium added to the corundum to produce the rutile ranges between about 0.06 and 0.2 atomic pct. In addition, about 1.4 pct of chromium is added as a pigmenting material to produce the red color of star rubies, and about 0.3 pct iron is added to produce the blue of star sapphires. The solubility of titanium in corundum at 2000°C appears to be limited to a maximum of a few atomic percent.

Boules containing precipitated rutile are turbid and show a sharply defined six-rayed star, equal or superior in quality to the best of the natural gems, when the boules are cut into properly oriented cabochon stones. A group of synthetic star sapphire gems is shown in Fig. 3. The rutile inclusions are clearly visible under moderate magnification and are found to be oriented in (0001) planes with their elongation parallel to the crystallographic a-axes of the corundum. A photomicrograph of the oriented, precipitated rutile inclusions in synthetic star sapphire is shown in Fig. 4. The identification of the inclusions as rutile has been proved by crushing the asteriated stones to a very small particle size in a ball mill and then separating the inclusions from the corundum by centrifuging the mixture in a heavy solution (Clerici solution), the specific gravity of which has been properly adjusted. Inclusions extracted in this way have been identified as rutile by x-ray methods together with optical and chemical tests. The asterism is due to the reflection and scattering of light by the oriented set of inclusions. Each ray of the visible star is perpendicular to a position of orientation of the inclusions. The amount of scattered light increases relative to the reflected light as the inclusions decrease in thickness to dimensions of the order of the wave-length of ordinary light. The scattering gives rise to the bluish and reddish borders that may be seen on the rays of the stars, the red side always away from the direction of incidence of the light, and to the overall reddish appearance of the star when the stone is viewed in transmitted light as compared to the bluish appearance in reflected light, the latter condition being that ordinarily obtained. The definition of the star also is influenced by the number of inclusions per unit volume, the degree of elongation and shape of the inclusions, and the orientation and perfection of polish of the cut stone. It may be noted that the precise nature of the inclusions is not of prime importance. The basic conditions that must be met to produce a star are only that the inclusions have an index of refraction different from that of the enclosing corundum, that they be elongate in shape, and that they be oriented. In some natural corundum the star or chatoyance appears to be caused by oriented sets of hollow tubules.

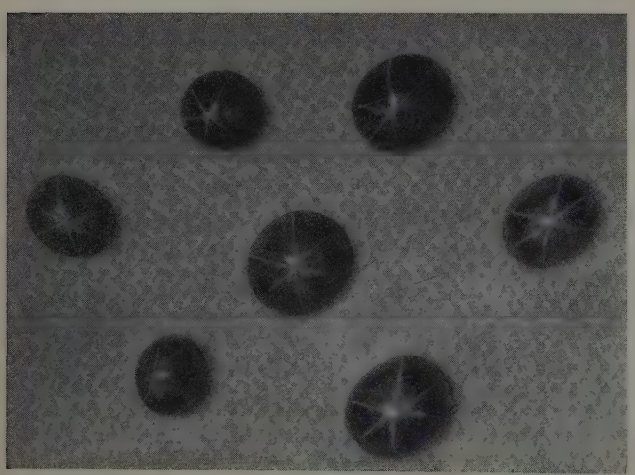


Fig. 3—Synthetic star sapphire cabochons, ranging in size from 10 to 20 carats.

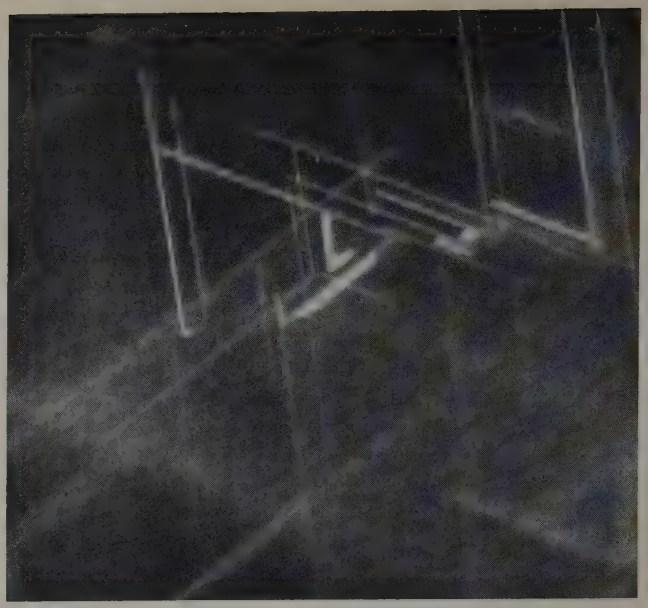


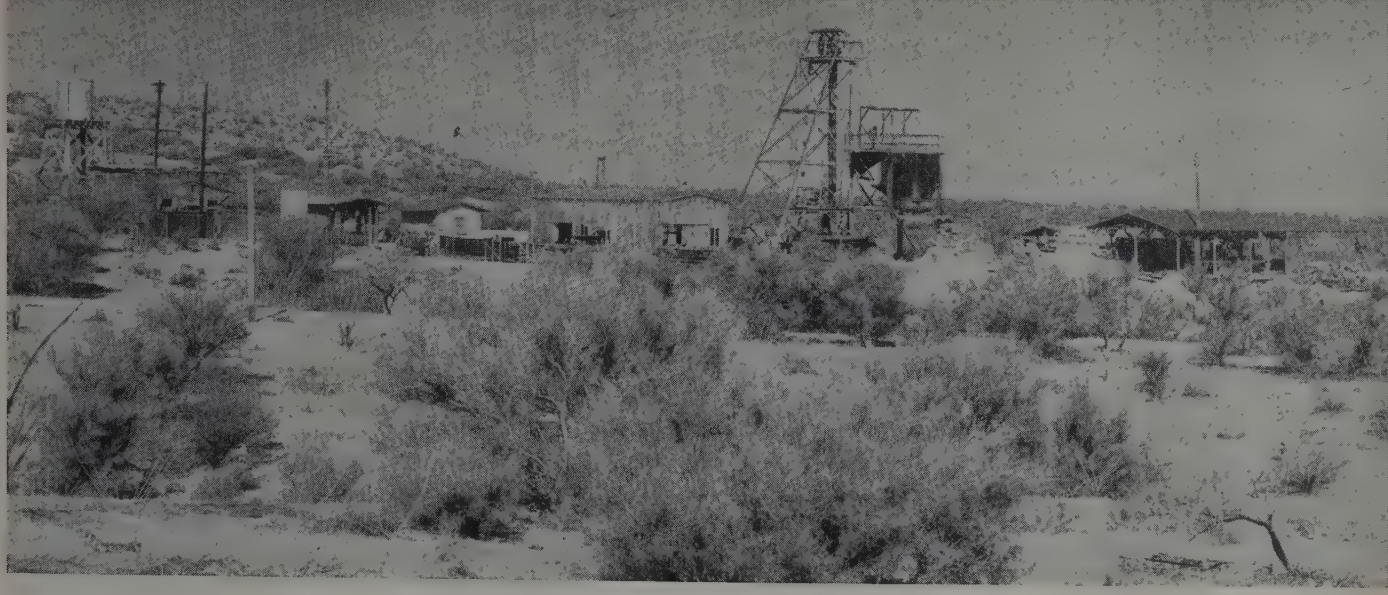
Fig. 4—Photomicrograph of oriented, needle-like rutile inclusions in synthetic star sapphire. The fishhook-like crystals are twins on (011). Real magnification about X1000.

The Linde process for the synthesis of asteriated corundum was patented in 1947, and commercial production was started in the same year. Unmounted stones currently sell commercially at a retail price of about $10 to $25 a carat depending on quality. They are ordinarily available in sizes running up to about 15 or 20 carats, but much larger stones have been grown.

References

[1] J. M. Güthe: Ueber den Astrios-Edelstein, Kl. 2. Denkschr. Kön. Baierischen Akad. Wiss., Münich, 1809. 76 pp.

[2] U. S. Patents 988,230 (1911), 1,004,505 (1911) and 2,488,507 (1949); German Patent 711,293 (1941).



Geophysical Discovery and Development Of The Pima Mine, Pima County, Arizona

A Successful Exploration Project

by Robert E. Thurmond,

Walter E. Heinrichs, Jr., and E. D. Spaulding

DISCOVERY of a commercial ore deposit has resulted from a program emphasizing geophysical techniques and concentrating upon exploration of alluvial-covered areas. United Geophysical Co. selected these areas as offering the best possibility of success, for here the surface evidence of ore deposits has been hidden from the eyes of prospector and geologist alike.

Pima mining district, Pima County, Arizona, was selected for initial exploration. The project was successful in that a commercial ore deposit was discovered and proved through drilling and extensive underground development.

A limited region of activity was chosen and library research was begun on available economic and regional geological publications relating to the mining districts. Field reconnaissance of those districts which seemed to hold exploration possibilities provided a general picture and ascertained the existence of alluvial-covered areas of interest. At the conclusion of the reconnaissance trips all data obtained were compiled, and the district which seemed to present the best exploration possibilities was selected by statistical methods. The statistical summary was based on the following considerations:

Geological Factors: 1—type of intrusive, 2—presence of favorable host rocks, 3—apparent intensity of mineralization, and 4—presence of favorable structure in covered or unexplored areas.

R. E. THURMOND, W. E. HEINRICHS, JR., and E. D. SPAULDING, Members AIME, are, respectively, Mine Superintendent, Pima Mine; Acting Manager of Mining Exploration; and Manager of Mining Exploration and Production, United Geophysical Co.

Discussion on this paper, TP 3746A, may be sent (2 copies) to AIME before April 30, 1954. Manuscript, Dec. 10, 1953. New York Meeting, February 1954.

Economic Factors: 1—past production from district or mine; 2—size, shape, grade, and type of ore deposit in the district; 3—ease of acquiring mineral rights; and 4—general development facility.

Exploration Favorability Factors: 1—results to be anticipated from geological methods alone; 2—application of geophysical methods, namely, topographic conditions, surface interference, depth-size relation, physical property contrasts as related to ores or controlling structural features; and 3—drilling factors.

Admittedly, many of these factors do not lend themselves to absolute evaluation, but relative evaluation, subject to inaccuracies of personal judgment, is always possible. A relative scale from 1 to 10 was established for every factor, representing varying degrees of favorability. For each district a numerical evaluation of each factor was established and a numerical total determined. The obvious advantage of this scheme is that a more concise summation can be presented by numerical means than can be achieved by descriptive means.

After a district had been selected as favorable, the first step was to carry out more detailed geological and economic studies to narrow down the areas of interest. The second step was to compile all available geological data from publications and add to these data by mapping where necessary. Geological interpretation and projection of structural or other control into covered or unexplored areas was made whenever logical. To determine the possibilities of geophysical application and to select a geophysical method, physical property contrasts were related to mineral assemblages and/or structural control.

If geophysics seemed applicable, trial profiles were run crossing the area normal to the strike and spaced

This page of Mining Transactions AIME follows p. 80. The intervening non-Transactions pages appeared in Mining Engineering.

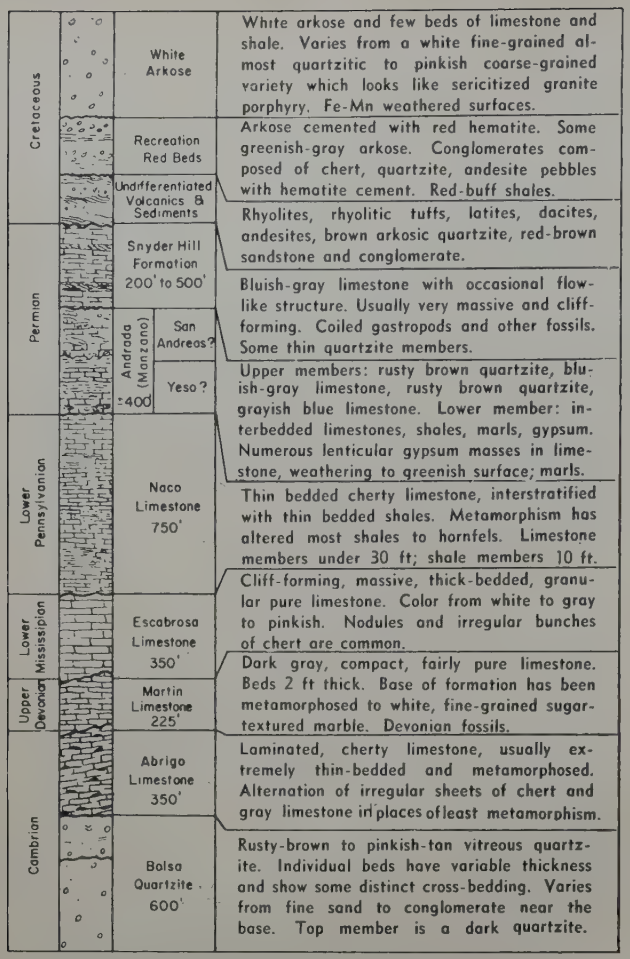

Cretaceous
Permian
Lower Pennsylvanian
Lower Mississippian
Upper Devonian
Cambrian
White Arkose
White arkose and few beds of limestone and shale. Varies from a white fine-grained almost quartzitic to pinkish coarse-grained variety which looks like sericitized granite porphyry. Fe-Mn weathered surfaces.
Recreation Red Beds
Arkose cemented with red hematite. Some greenish-gray arkose. Conglomerates composed of chert, quartzite, andesite pebbles with hematite cement. Red-buff shales.
Undifferentiated Volcanics & Sediments
Rhyolites, rhyolitic tuffs, latites, dacites, andesites, brown arkosic quartzite, red-brown sandstone and conglomerate.
Snyder Hill Formation 200' to 500'
Bluish-gray limestone with occasional flow-like structure. Usually very massive and cliff-forming. Coiled gastropods and other fossils. Some thin quartzite members.
Andrada (Manzano) ±400'
San Andreas?
Yeso?
Upper members: rusty brown quartzite, bluish-gray limestone, rusty brown quartzite, grayish blue limestone. Lower member: interbedded limestones, shales, marls, gypsum. Numerous lenticular gypsum masses in limestone, weathering to greenish surface; marls.
Naco Limestone 750'
Thin bedded cherty limestone, interstratified with thin bedded shales. Metamorphism has altered most shales to hornfels. Limestone members under 30 ft; shale members 10 ft.
Escabrosa Limestone 350'
Cliff-forming, massive, thick-bedded, granular pure limestone. Color from white to gray to pinkish. Nodules and irregular bunches of chert are common.
Martin Limestone 225'
Dark gray, compact, fairly pure limestone. Beds 2 ft thick. Base of formation has been metamorphosed to white, fine-grained sugar-textured marble. Devonian fossils.
Abrigo Limestone 350'
Laminated, cherty limestone, usually extremely thin-bedded and metamorphosed. Alternation of irregular sheets of chert and gray limestone in places of least metamorphism.
Bolsa Quartzite 600'
Rusty-brown to pinkish-tan vitreous quartzite. Individual beds have variable thickness and show some distinct cross-bedding. Varies from fine sand to conglomerate near the base. Top member is a dark quartzite.

Fig. 1—Generalized columnar section, Mineral Hill area.

at wide intervals. Where possible, simple, rapid methods were first employed, after which the interesting areas were detailed. When a second geophysical method seemed applicable, the areas were re-surveyed. If the anomalous areas were apparent from results of two or more independent methods, interpretation and drilling considerations were facilitated. Since a reasonable geological hypothesis was already established, the geophysical interpretation consisted primarily of an estimation of horizontal and vertical location of the drilling target. This interpretation was checked against geological thinking to establish consistency in both sets of data.

Location and General Description

By the process of elimination just described the Pima mining district was chosen from some 30 districts. The Mineral Hill area where Pima mine is located was selected as one part of the Pima district to be investigated.

The Mineral Hill area is in the northeastern part of the Pima district, Pima County, Arizona, 20 miles southwest of Tucson. The district includes that part of the Santa Cruz river valley which is also the eastern slope of the Sierrita mountains. Most of the district is shown on the U. S. Geological Survey, Twin Buttes Quadrangle. Topography varies from flat to rugged, with numerous dry wash gulches. The usual brush, desert trees, and cactus cover the terrain, see frontispiece. Most of the area is accessible by good dirt roads and trails.

Past development in the Pima district followed the typical pattern of many western mining districts. Early production was in silver and copper. During this period two small smelters or furnaces were built at Sahuarita and Mineral Hill. Production continued until shortly after World War I, with a gradual decline to total inactivity by 1929. During World War II activity was resumed, significant lead-zinc-copper production coming from the San Xavier and other smaller properties.

At present, in addition to the Pima mine development, the Banner Mining Co. is developing its holdings and constructing a mill at Mineral Hill. Total production for the district is valued at over $20 million in copper, lead, zinc, silver, gold, and molybdenum.

Geology

The Sierrita Mountains are the most prominent physiographic feature of the Pima district. The northern portion of this range is the topographic expression of a batholithic mass of intrusive granite which comprises the main regional geologic structure of the district. Small sedimentary blocks of extremely complicated structure are exposed along the lower flanks of the range. Sedimentary structure is oriented east-west, roughly normal to the long dimension of the Sierrita range. Alluvial fans resulting from recent erosion of the range form a blanket of cover which overlaps the lower slopes of the mountains. In general, only the sedimentary blocks, particularly of limestone, have been resistant to erosion and so remain as hills surrounded by a blanket of alluvium. On the lower slopes where igneous rocks are exposed erosion has been greater.

The granite intrusion is thought to have occurred during the late Laramide time, although in some locations the coarse-grained texture of the igneous rocks and the lack of intense metamorphism along the sedimentary-igneous contacts has suggested the possibility of pre-Cambrian granite as well as younger intrusive granite.

In places both granites and sediments have been cut by later dikes which appear in a wide range of sizes, shapes, and composition. Volcanic rock which varies from rhyolite to basalt may be found in portions of the district. These are thought to be pre-Cretaceous, although some may be of a later period.

Sedimentary rocks exposed in the district range in age from Cambrian through Cretaceous. Generally speaking, these rocks may be correlated with established geological columns throughout the southwest. Fig. 1 is a graph representing the geological column.

Important ore deposits of the district can be fitted into the broad category of contact metamorphic deposits. In specific cases their characteristics vary, depending on their proximity to the related intrusive masses.

The Mineral Hill deposit is the only known deposit in the immediate vicinity of the Pima mine. The outcrop nearest Pima mine is exposed about a quarter mile west on Mineral Hill. Since the structural relations of Mineral Hill were a lead to the exploration of the alluvial-covered Pima area, a brief general description of the geology, structure, and ore relationships is pertinent, see Fig. 2.

Mineral Hill consists of two blocks of sediments separated by a major thrust fault striking nearly east-west and dipping approximately 50° to the south. These sediments form a small group of hills surrounded by alluvium except on the west side, where granite outcrops and is in contact with the

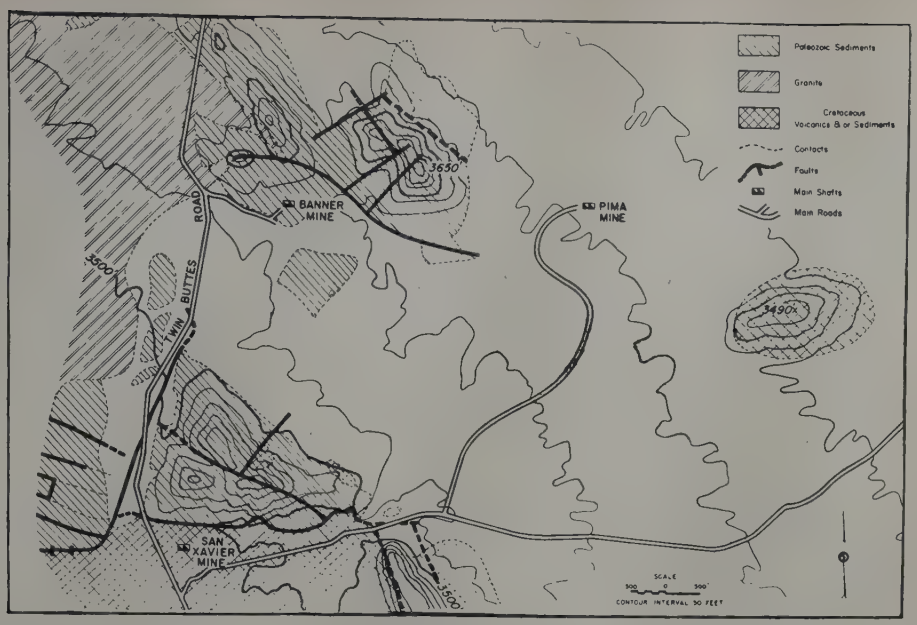


Fig. 2—Surface and general geology of the Mineral Hill area, Pima district, Arizona.

sediments. The western block of sediments is a reversed sequence with Cambrian, Devonian, Pennsylvanian, and Permian formations exposed from southwest to northeast and dipping steeply to the south. Much cross faulting results in complex structural relations within this block. South of the major fault Pennsylvanian and Permian sediments crop out in normal sequence, dipping steeply to the south; however, the structure here is somewhat obscured because of the alluvial cover on the lower slope of the hill.

The major thrust fault zone may have formed a channel along which igneous rocks have intruded the sediments. The ore deposits occur in favorable sedimentary members along the fault zone and in close proximity to the intrusive rocks. They are typical contact metamorphic deposits in that there is a normal contact-zoning of the high temperature gangue and ore minerals. The ore deposits are irregular in grade, shape, and distribution. The chief ore mineral is chalcopyrite, although locally bornite and chalcocite are present. In addition to the copper, minor amounts of zinc, molybdenum, and tungsten occur throughout the orebodies. Locally magnetite and pyrrhotite are present in varying quantities in close association with the ore minerals.

The apparent primary control of the deposits at Mineral Hill was the major thrust fault zone striking east-west. It was logical to assume that east of Mineral Hill there might be, under cover of alluvium, a continuation of this fault which would again cut favorable sediments. Possible blind ore deposits made this a good area for geophysical prospecting.

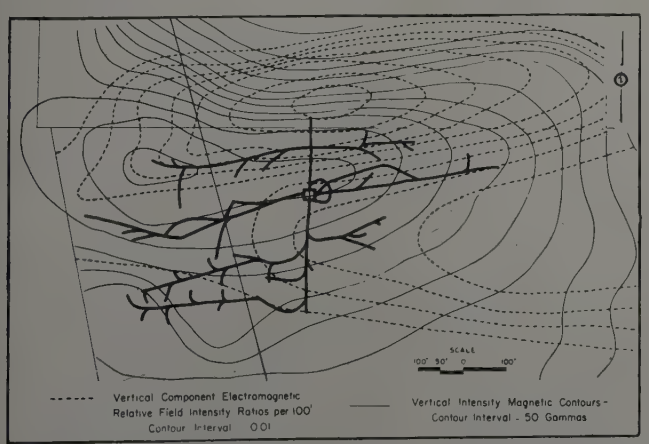

SCALE
100' 50' 0 100'
Vertical Component Electromagnetic Relative Field Intensity Ratios per 100' Contour Interval 0.01
Vertical Intensity Magnetic Contours - Contour Interval - 50 Gammas

Fig. 3—Relationship between geophysical anomalies and the mine workings.

Geophysical Work

Investigation of known deposits in the district indicated that ore minerals, along with some associated gangue minerals, might possess sufficient physical property contrast with the country rock to support geophysical applications in the Pima district. To establish the magnitude of physical property contrast between the different rocks, in-place samples were taken from the surface and from accessible underground workings. The samples were tested for magnetic susceptibility, density, and conductivity. By means of these data and the size and shape of known orebodies, theoretical results to be expected from geophysical work were computed.

Results indicated that zones of magnetic concentration of the size expected could be detected with the magnetometer to a depth of 200 to 300 ft. Since much of the ore had been found in association with these zones, it was decided to make limited field tests with the magnetometer first, and if these were successful, more extensive surveys by other methods.

Preliminary magnetic tests were made by running long profile lines. A standard temperature-compensated, vertical intensity magnetometer was used. Readings were taken along the profile line every 12.5 ft, 25 ft or 50 ft, depending upon the rate of change between successive readings. Results of these tests were favorable in the Mineral Hill area and a detailed survey was started.

In this work stations were located with respect to a grid system. All readings were related to one main base station which was occupied each morning and evening. The overall accuracy of each observation, considering all errors, was no greater than plus or minus six gammas.

One important magnetic feature was delineated off the alluvial-covered eastern flank of Mineral Hill. This was called the Alpha area and was the site where prior geologic interpretation had indicated favorable possibilities, see Figs. 2 and 3. The magnetic high compared favorably with results obtained over other deposits that had demonstrated a close magnetic relationship to known mineralization.

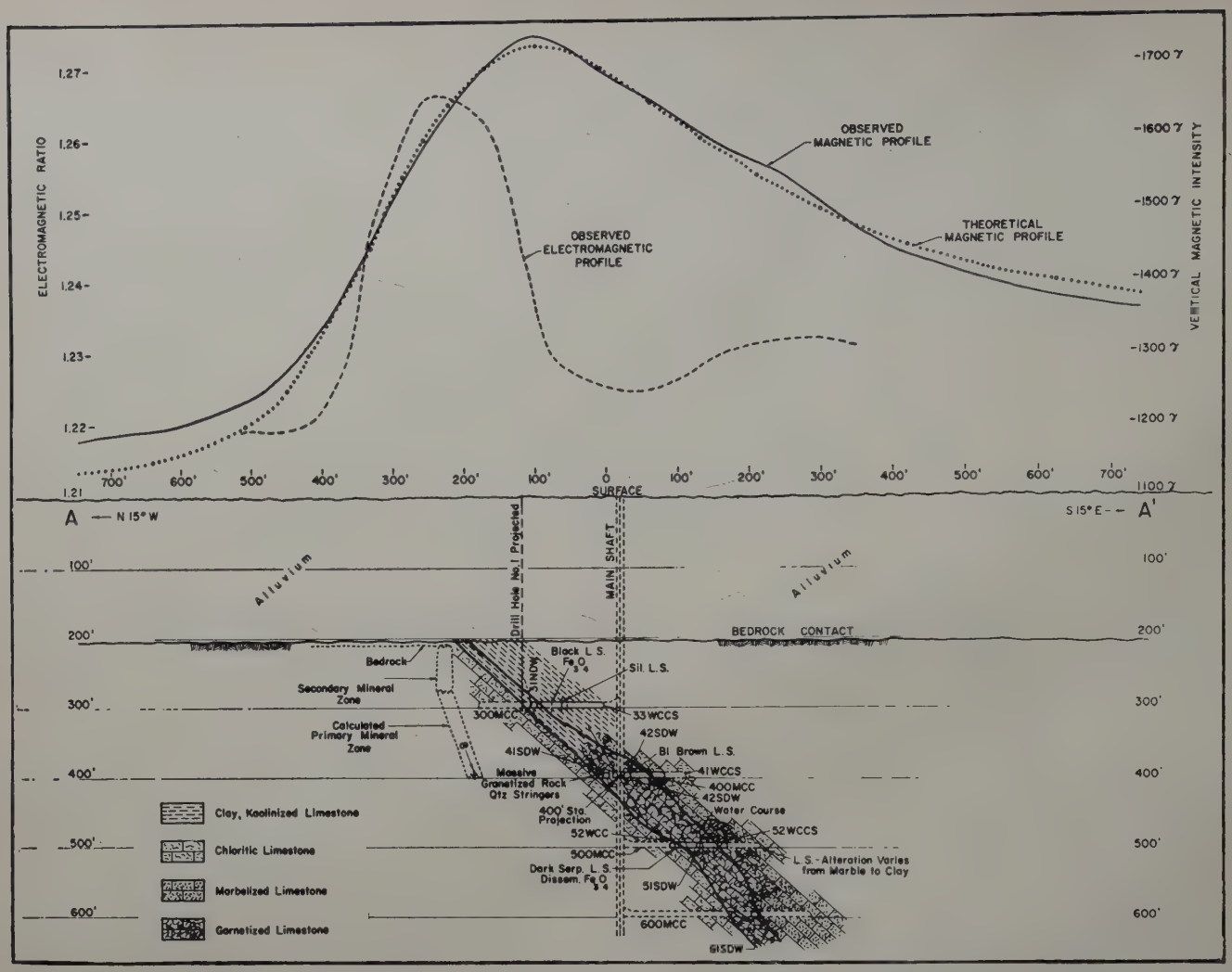


Fig. 4—Geophysical profiles and geologic section through A-A'. Geophysical bedrock and secondary mineral zone were estimated.

However, it was known that magnetite could occur without ore and that further information was desirable before a drilling program was planned. Investigation of the known mineral occurrences had also shown good electrical and density contrasts in addition to the magnetic properties. A study of the Alpha magnetic anomaly indicated a depth to the upper surface of the orebody of 210 ft, plus or minus 25 pct, see Fig. 4. This order of magnitude depth was confirmed in general by geological extrapolation and resistivity depth probing. Actual depths determined from drilling ranged from 205 to 215 ft. Calculations disclosed that work to this depth would be beyond the marginal limits of good gravity results, even with strong density contrasts. This suggested an application requiring an electrical method with penetration and resolution to at least 300 ft deep. Experience indicated that the electromagnetic method would be the most feasible. The required equipment was designed according to conventional principles and established procedures already used in the industry. Improved instrumentation provided maximum accuracy and dependability of results.

Field use of this equipment involved laying out a long wire grounded at each end, connected to a 400-cycle generator. Electrical measurements were made by comparing the electromagnetic field at two search coils across an electronic bridge with high amplification. Results of these measurements were plotted as relative field intensity ratios or phase angle differences in typical profile or contour form. An examination was then made of the areas of abnormal electrical gradient effects which would correlate with zones of unusual conductivity.

In the Alpha area an electromagnetic high was obtained which compared very well with the magnetic high, see Fig. 3. This evidence served to justify running a few experimental gravity profiles. Gravity results did not directly reflect a possible ore zone, but they did show an abnormality, probably due to basement structure, in the same location. Following the gravity work, a natural potential survey was made over the anomalous zone. This indicated a moderate oxidation area which coincided with previous information except that the center of highest intensity was offset to northwest. This was caused by more mineralization above the water table in that direction and by thinning of alluvial cover.

In compiling of geophysical data, corrections were kept to the minimum necessary to relate the observed values. Final interpretation was made by directly comparing the profiles or contour maps of results obtained from each method.

Summary of Surface Exploration: For the initial drilling, reliance was placed on the magnetic and electromagnetic results. Location of the first drill hole was based on an analysis of the magnetic data. The electromagnetic data confirmed this interpreta-

tion. Before drilling, geophysical analysis suggested that the orebody was a lenticular-shaped deposit dipping steeply to the south and striking nearly east-west. It was estimated that the upper surface of the ore was approximately 200 ft deep.

The first drill hole encountered bedrock and oxidized mineralization at 209 ft and sulphide ore at 255 ft. The samples of rock below the bedrock surface were highly magnetic and good conductors.

Development Program

Surface Drill Exploration: Upon completion of the first drill hole it was found that assays warranted a systematic drilling program to determine extent of mineralization and to provide sufficient knowledge for further interpretation of geophysical data. The first drill hole did not indicate a commercial deposit, but it did show the presence of copper mineralization. It should be noted that little or no prior information was available to guide the exploration, but the interpretation of the geophysical results was correct in direction of dip, depth of overburden, and general strike. Location of the second drill hole was guided from information obtained from the first, and all other holes were located by means of the magnetic contour map.

The pertinent problem required drilling through at least 200 ft of alluvium over the sub-outcrop. It was decided that standard diamond drilling practice in the alluvium would be impractical, but if oil field drilling methods were used, progress would be more rapid. The desired drill in the form of a Joy 22-HD was not available, but a Republic shot hole drill with a chain pulldown could be obtained.

Two strings of tools were used with the drill. To drill through the alluvium the tools used were 2⅜-in. diam drill pipe with a 3-in. diam drill collar and a Hughes W7R 3⅞-in. diam rock bit. Casing was set and cemented in the bedrock at any convenient hard strata. All coring was done with AX core barrels. To adapt the A rod for use on the Republic drill, a square kelly was fabricated with a chuck suited to the A rod. The kelly was fed through the rotary table by gravity, as no positive feed was available in the form of screw or hydraulic mechanism. This crude rigging was used until a Joy 22HD drill was purchased. The equipment mounted on a Ford, Marmon Harrington four-wheel drive truck consisted of the drill and a 4½x6-in. mud pump powered by the truck engine. The pump was rigged for use as a mud pump or as a clear water pump required when coring. This was accomplished by use of a bypass piped into the suction line. The multiple-purpose feature of the drill was successful in drilling either vertical or angle holes. It was not unusual to drill 200 ft to bedrock and set casing in one 8-hr shift. Eighteen holes were drilled, 16 completed, and 2 abandoned because of drilling difficulties before the mineralized zone was cut. Total footage drilled was about 7200 ft.

Underground Development: From the results of the drilling, a report was prepared recommending the sinking of an exploration shaft to 400 ft to explore the ore zone on the 300 and 400 levels. In November 1951 Pima Mining Co. was incorporated to carry out the program. Surface construction of the mine surface plant began in December. A shaft contractor was engaged to begin work on Jan. 1, 1952. Actual sinking was underway by Jan. 15. There was difficulty sinking through the 200 ft of alluvium, but nothing to retard progress seriously.

By May 1952 the 400 level station was cut. Lateral development on both the 300 and 400 levels started immediately following completion of the shaft stations. Two drifts driven east and west on the 400 level from the main crosscut explored the structure laterally for a distance of 925 ft. Concurrently the 300 level was explored laterally for a distance of 610 ft east and west of the main crosscut.

Following the exploration on two levels it was decided to sink to the 600 level. Nine holes were drilled from the 400 level, cutting the structure at varying depths. The deepest hole intersected the mineralized zone approximately 200 ft below the 400 level. Results from the nine holes encouraged further sinking of the shaft. Sinking commenced on Nov. 15, 1952, and by April 1953 the shaft was complete with stations at the 500 and 600 levels. This also included a permanent pump station on the 600 level capable of handling 1000 gpm.

Development of both lower levels was carried on concurrently, and by Sept. 1, 1953, the 500 level was explored laterally for a distance of 600 ft, the 600 level laterally for a distance of 400 ft. Lateral de-

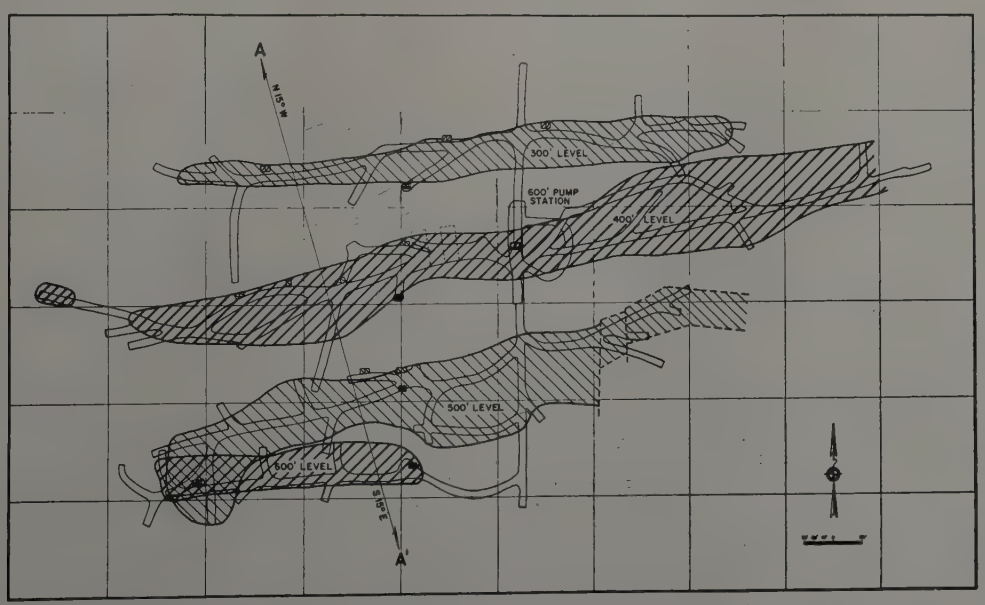


Fig. 5—Horizontal outline of ore shoots, all levels.

velopment on the lower levels is still in progress, although no work has been initiated east of the main crosscut on the 600 level. Preliminary diamond drilling has indicated a probably large volume of water in this section, and no work is proposed until the country drains.

Present activity is concentrated on vertical development and stope development. The width of the orebody and the ground characteristics require a stoping method dependent upon support and fill.

Experimental steps are in progress to prepare sections for a Mitchell slice system, and where ground conditions permit, a panel cut-and-fill system. Further work is required before a definite mining plan can be developed.

Underground Geology and Description of Orebody: The subsurface contact between the alluvial fill and country rock represents the suboutcrop of the orebody. It has been agreed that the section found in the mine consists of Paleozoic sediments and is separated from the Mineral Hill area by a possible north-south fault.

Degree of alteration varies widely throughout the mine. The mineralized zone is intensely altered, the alteration extending into both the hanging wall and footwall and decreasing in intensity perpendicular to the strike. The footwall proper consists of greenish-black chloritic material altered from limestone. The gangue material within the ore zone and locally on the hanging wall, particularly on the 300, 400, and 500 levels, is an intensely altered garnetized limestone. The garnetized limestone is an expression of alteration, but where the rock has been completely kaolinized alteration has been intense. The kaolinized material suggests the presence of porphyry, as there is some complete digestion with remnant limestone fragments included. Less intense alteration is expressed in the form of marbleized limestone. These instances are local and do not represent the general alteration pattern.

The degree of alteration normal to the walls varies widely in thickness from 20 to 100 ft or more. Ore mineralization is directly related to degree of alteration. Concentration of ore is associated with the garnet and kaolin zones.

The mineral assemblage represents that typically found in contact metamorphic deposits. Chalcopyrite is the principal ore mineral. Above the 300 level considerable secondary mineralization is present as chalcocite and native copper. The gangue minerals are epidote, garnet, magnetite, and calcium silicates, which are found throughout the orebody.

Mineralization and alteration are within a structural break that controls the deposition. Replacement is evident, but the fault zone appears to be the controlling factor. Cross-fracturing is present, but whether or not it is of importance is not known.

The orebody may be likened to a porphyry with phenocrysts of varying size representing the high-grade chalcopyrite, the matrix composed of intensely altered limestone containing chalcopyrite in disseminated form. The massive chalcopyrite is scattered and irregular in spacing and size. The localizing of the higher grade zones is not clearly understood, but a favorable limestone bed is probably a controlling factor. Alteration generally has destroyed any original identity, although a dark limestone present appears to be the favorable host rock.

Lateral and vertical development has not limited the extent of the deposit, although the lateral extent on the 400 level appears determined. The property line to the west limits exploration in that direction, and although mineralization weakens on the west side, the adjoining property has uncovered a possibly related orebody. Present exploration is concentrated to the east of the shaft, as little other work can be done to the west because of property limitations. Fig. 5 shows the width and lateral extent of the ore zone at the various levels.

Production and Future Plans: Shipments of ore from the mine commenced in May 1952 and have been continuous with the exception of a period between December 1952 and May 1953. Twenty-six thousand tons of ore containing 3,173,000 lb of copper have been shipped to the American Smelting and Refining Co. smelter at El Paso. Ninety percent of ore shipped was produced from mine development and 10 pct from stope development. Average grade of ore shipped, calculated from smelter settlement sheets, approximated 6 pct.

The orebody is of commercial milling grade, and sufficient tonnage of mill grade ore is in sight to warrant construction of a medium-sized concentrator. In conjunction with the mill, plans would require a new shaft, a surface plant, and preparation of the mine to deliver a minimum 350 tons a day.

The Pima mine is another example of successful application of mining geophysical methods. It is believed that its discovery is only a forerunner of the development of other hidden orebodies. Work at the Pima mine will not be completed until it becomes a profitable metal producer of the Southwest.

Acknowledgments

The authors are grateful for the support and guidance rendered by Herbert Hoover, Jr., Raymond A. Peterson, and M. G. Edwards throughout the project. They also appreciate the suggestions and advice given by B. S. Butler in the initial exploration phase and the interpretations made by Eldred Wilson of the geological problems.

References

F. L. Ransome: Ore Deposits of the Sierrita Mountains, Pima County, Arizona. U. S. Geol. Survey, *Bull. 725* (1922) pp. 407-428.

E. R. Gordon: The Geology of the Twin Buttes Mining District. University of Arizona thesis (1922) 10 pp.

R. L. Brown: Geology and Ore Deposits of the Twin Buttes District. University of Arizona thesis (1926) 40 pp.

C. F. Park, Jr.: Geology of the San Xavier District. University of Arizona thesis (1929) 30 pp.

B. N. Webber: Marcasite in the Contact Metamorphic Ore Deposits of the Twin Buttes District, Pima County, Arizona. *Economic Geology* (1929) **24**, pp. 304-310.

E. B. Eckel: Geology and Ore Deposits of the Mineral Hill Area, Pima County, Arizona. University of Arizona thesis (1930) 51 pp.

Eldred D. Wilson: Tungsten Deposits of Arizona. Arizona Bur. Mines, *Bull. 148* (1941) pp. 36-37.

M. N. Mayuga: The Geology and Ore Deposits of the Helmet Peak Area, Pima County, Arizona. University of Arizona thesis (1942) 124 pp.

M. J. Elsing and R. E. S. Heineman: Arizona Metal Production. Arizona Bur. Mines, *Bull. 140* (1936).

H. A. Whitcomb: Geology of the Morgan Mine Area, Twin Buttes, Arizona. University of Arizona thesis (1948) 82 pp.

Eldred D. Wilson: "The Pima District" in Arizona Zinc and Lead Deposits, Part I. Arizona Bur. Mines, *Bull. 156* (1950) pp. 39-51.

J. B. Cummings and T. M. Romslo: Investigation of Twin Buttes Copper Mines, Pima County, Arizona. U. S. Bur. Mines, *R. I. 4732* (1950) 12 pp.

American Potash & Chemical Corp. Main Plant Cycle

by M .L. Leonardi

THE Searles Lake orebody is located in the northwest corner of San Bernardino County. It is a dry lake bed with an exposed salt surface covering an area of 12 square miles. Recoverable mineral values are contained in the mother liquor below the surface of the lake. Stratification in the lake bed has separated the brine into two bodies which differ in composition. Although liquor is processed from both bodies, this paper will discuss only the upper structure brine.

Fig. 1 illustrates a typical cross-section of the two commercial orebodies. The orebody is composed of a porous salt deposit 70 to 90 ft deep. The upper structure is separated from the lower orebody by a 12 to 16-ft thick impervious mud seam, as shown in Fig. 1. These salt structures are composed of 55 pct solid-phase salts and 45 pct voids which are filled with the original mother liquor.

The brine wells are drilled to the separating mud seam and cased to within 10 ft of the bottom. This is done to draw the brine horizontally from the bottom of the structure. It is pumped with multistage centrifugal pumps into the plant at the rate of 3 million gal per day.

The first process that was successful was developed by Charles P. Grimwood for the recovery of potash. The first evaporator unit was built in 1916. In the early twenties, Dr. Morse worked out a process for the recovery of borax. This made the cycle more efficient, as the end liquor could be sent back to the evaporators rather than being sewered. In 1926 the American Potash & Chemical Corp. was formed as a new company, and the entire plant was remodeled. The plant at that time produced only potash, borax, and boric acid. Since then the American Potash & Chemical Corp. has added processes for the production of USP boric acid, refined potash, sulphate of potash, soda ash, salt cake, lithium concentrates, Pyrobor ($Na_2B_4O_7$) bromine, phosphoric acid, and lithium carbonate.

The main plant cycle may be depicted as a closed cycle, see Fig. 2. The raw material, brine, enters the cycle to be mixed with the end liquor, known as ML2, from the pentahydrate borax crystallizers. The mixture of these two forms evaporator feed. Evaporator feed is pumped to the evaporators where it is concentrated, with respect to potash and borax. In the same operation water vapor, sodium chloride, salt trap salt, and clarifier salt are removed from the cycle, see Fig. 3 for potash plant product.

The evaporators produce a concentrated liquor which contains approximately 19.5 pct KCl. This liquor is diluted as it enters the potash plant to keep all salts, except potash (KCl, 97.0 pct) in solution. Here the moist potash leaves the cycle at 100°F. The end liquor, known as ML1, is pumped to the borax pentahydrate crystallizers, where crude borax pentahydrate is crystallized and removed as solid phase.

M. L. LEONARDI, Member AIME, is Crystallizer Superintendent, American Potash & Chemical Corp., Trona, Calif.

Discussion on this paper, TP 3728H, may be sent (2 copies) to AIME before April 30, 1954. Manuscript, Jan. 2, 1953. Los Angeles Meeting, February 1953.

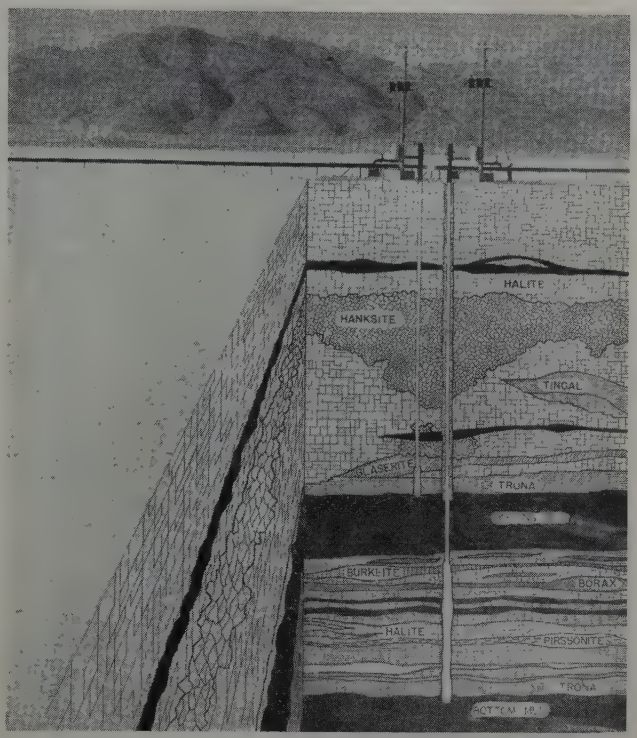


Fig. 1—Cutaway of Searles Lake salt structure.

The ML2 is sent back to pan feed to be reconcentrated, see page 207.

Note that the only water to leave the cycle is in the form of vapor and moisture in the solid phase products crystallized. Thus there is a constantly cycling volume of liquor to which brine is added. Since the volume of liquor cycled does not increase, the brine is, in effect, evaporated to dryness. This would be true if there were no liquor losses. But, as in all processes, there are always unavoidable and accidental losses which reduce the volume of cycling liquors. The losses must be made up with brine.

The concentration process is the beginning and the end of the cycling liquors. In this process there are three evaporator units of the triple effect countercurrent type, that is, there are three pans in each unit and the heat flows in one direction while the liquor flows the other way through the evaporator pans, see Fig. 4.

During the evaporation process a great deal of sodium chloride, burkeite, some sodium carbonate monohydrate, and a little lithium-sodium phosphate are crystallized. The volume of these salts is so great that they must be removed as they are formed or the process would come to a standstill.

Brine and recycled mother liquor No. 2 enter the third effect evaporator pan from the evaporator feed storage tanks, see Fig. 5. A steady flow of liquor is removed from the bottom of the No. 3 pan and is pumped through the No. 3 cone of the salt trap, a clear liquor being returned to the No. 3 pan. A portion of this clear liquor is pumped to the second effect pan. This process is repeated in each pan. The liquor from the No. 2 pan is pumped through the No. 2 salt trap cone and returned to the No. 2 pan.

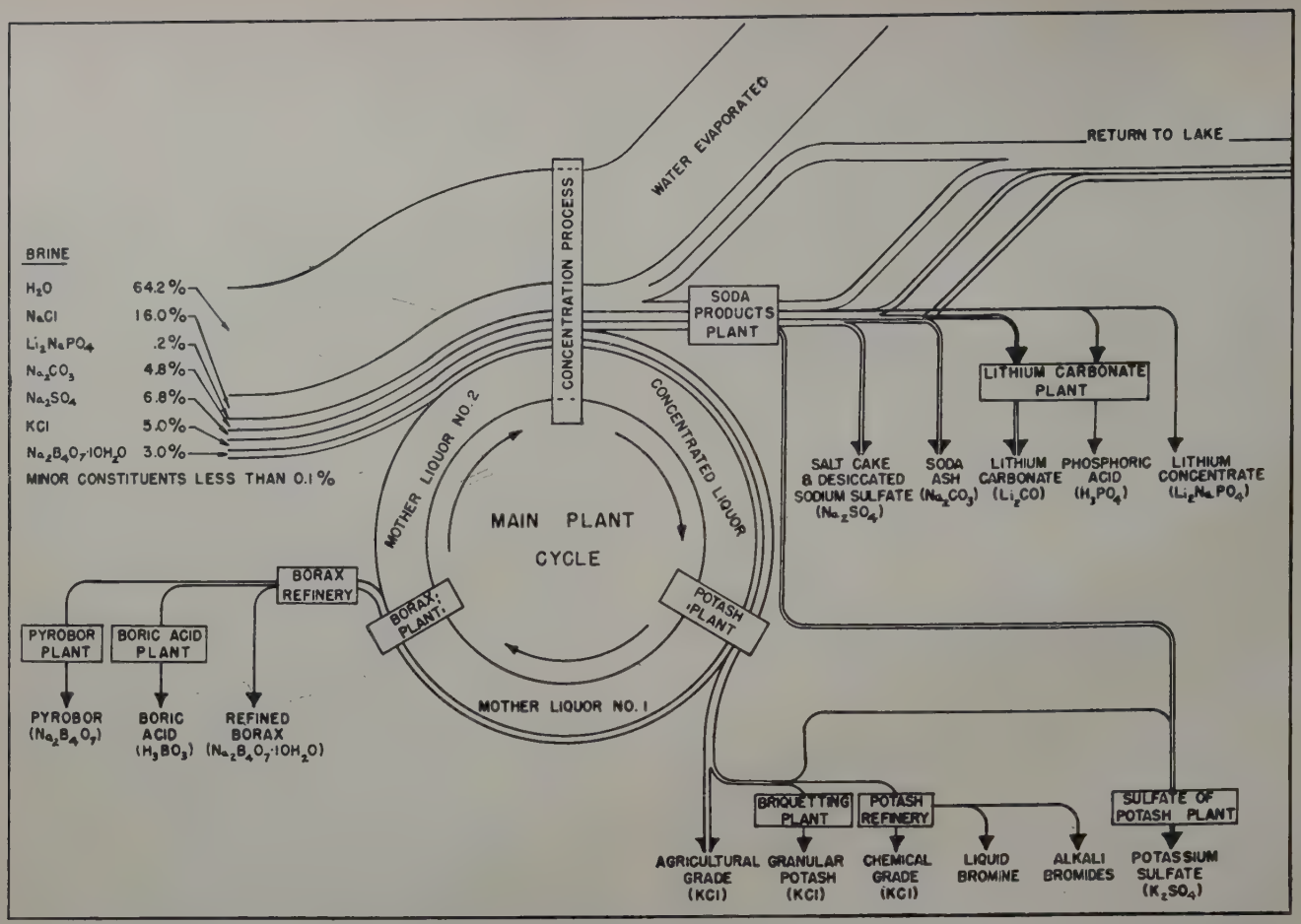


Fig. 2—Evaporator feed pumped to evaporators is concentrated, with respect to potash and borax, and in the same operation water vapor, sodium chloride, salt trap salt, and clarifier salt are removed from the cycle.

A portion of this liquor is advanced to the No. 1 pan. Finally, the liquor in the No. 1 pan is pumped to the No. 1 cone of the salt trap and clear liquor returned to the No. 1 pan. A portion is advanced to the clarifier cone, the amount being controlled by the KCl concentration in the liquor. It is desired to maintain a 36.5 g of KCl per 100 g of water.

The clarifier cone overflow is pumped to a 65-ft thickener. The clear overflow from the thickener is sent to two large storage tanks at the potash plant. The underflow from the salt trap is sent to a hydraulic classifier where the burkeite is separated from the sodium chloride. The underflows from the clarifier cone and concentrated liquor thickener contain burkeite, sodium carbonate monohydrate, lithium concentrates, and approximately 18 pct sodium chloride. These salts are sent to two 6x8-ft Oliver vacuum filters. To prevent flashing in the filters the temperature is reduced to 200°F by mixing with ML2. The salts are sent to the soda products system for processing and the filtrate is returned to the No. 1 pan for reconcentration.

The amount of concentrated liquor advanced to the clarifier cone determines the amount of pan feed pumped to the evaporators. It is necessary to maintain pre-determined levels in all the evaporator pans for proper operation. This is accomplished by transferring liquor ahead from the third-effect pan and controlling the level in the third-effect pan by feeding evaporator feed from the storage tanks.

The solid phase salts in the liquors from the evaporator pans are settled and separated from the liquors in the respective cones of the salt traps. Since the cones are superimposed in one vessel and are open at their apices, the combined salts leave through the bottom of the salt trap. This salt is either sent to 6x8-ft Oliver filters and then to a hydraulic classifier, or directly to the classifier. In either case the entrained brine is sent back to the No. 3 pan.

In the operation of the salt traps it is undesirable to return liquor from the first-effect pan which is highly concentrated to the third-effect pan where the liquor is weak. The salt trap is constructed to minimize this recirculating load. The liquor accompanying the salt as it enters the No. 2 cone from the No. 1 cone of the trap is displaced (or elutriated) from the salt by a flow of clear liquor from the No. 2 cone, which is returned, in part, to the No. 1 pan. This process of elutriation is repeated in each cone of the trap by pumping of the liquor from the cone below into the stream of advancing salt to produce a countercurrent flow, the salt down and the liquor up, in the same manner that the liquor is advanced in the evaporator pans.

The salt trap is rather small and will not separate the major part of the fine salts, namely, burkeite ($2Na_2SO_4 \cdot Na_2CO_3$), sodium carbonate monohydrate, and lithium-sodium phosphate present in the evaporator liquors. As a consequence these materials travel on through and are settled in the clarifier cone and the concentrated liquor thickener. The underflows are pumped to 6x8-ft Oliver filters where the salts are removed from the liquor. The liquor is returned to the system and the salts are pumped to the soda products plant.

The losses in the evaporators are of three types: lost evaporation time, liquor losses, and dilution of

the liquor with water which must be evaporated. The evaporators must be regularly boiled out and the heaters washed out on schedule. These operations cause losses in all the above categories, though with careful supervision and improved techniques the losses are reduced to a minimum.

There are losses also at the filters, since it is impossible to remove all the entrained liquor from the salts being removed from the system. These filter cakes contain 12 to 16 pct moisture. The problem is one of balance; if the liquor is completely removed, too much wash medium enters the system. Brine is used to displace the liquor in the salt trap salt, since any brine entering the system becomes evaporator feed, and this filtrate is returned to the No. 3 pan.

In this triple effect evaporator system the liquor flows countercurrent to the heat. The path of the liquor and salt has been previously described. The reader may now follow the path of the heat through the system, see Fig. 6.

Heat enters the system in the first-effect heaters as 35-lb steam exhausted from the steam turbines in the powerhouse. The liquor is pumped from the bottom of the evaporator pan through four-pass external heaters and is returned to the pan near the surface, releasing water vapors. These vapors pass through separators, which remove any entrained liquor, and are sent to second-effect heaters where they heat the liquor in the second-effect pan. The liquor in this pan boils, releasing vapors which are used to heat liquor in the third-effect pan. Finally the vapor in the No. 3 pan is drawn to a barometric condenser and is condensed in the circulating spray pond water. The water in the brine thus leaves the system as condensate in the second and third-effect heaters and in the barometric tail water.

The humidity in Trona is normally low; therefore the barometric condenser water is pumped over induced draft towers. Since the vapor pressure of water in the atmosphere is below the vapor pressure of water at the temperature of the tail water, the latter will evaporate, giving up heat until the temperature has gone down and the vapor pressure approaches that of the atmosphere. The old natural draft towers have been replaced by induced draft types which are much more efficient.

In this concentration process 5 pct potash brine is combined with 11.5 pct potash ML2 to yield about a 7.5 pct potash evaporator feed, to produce about a 19.5 pct potash concentrated liquor, see Table I.

Here is a typical concentrated liquor analysis:

KCl	19.5
KBr	1.1
$Na_2B_4O_7$	10.0
Na_2CO_3	6.3
Na_2SO_4	1.6
Na_2S	0.7
P_2O_5	0.6
NaCl	7.0
H_2O	52.7

Though losses are undesirable in the system, they do prevent other salts from reaching concentrations that might interfere if they were not removed. One measure of the losses in the plant is the concentration ratio of some of these minor constituents in the brine. If the brine were evaporated to dryness they would precipitate; therefore it is obvious that they must have been lost while they were still in solution. Since liquor is lost from the cycle, the brine must be evaporated to the point where its volume equals the volume of the liquor lost. The I_2 concentration ratio is used to indicate the liquor losses. This ratio varies from 28 to 36, which means that the brine is concentrated until 1/28 to 1/36 of the original water is left before it is lost.

In these triple-effect evaporators, the evaporator feed enters the third effect at 125°F and boils at about that temperature. However, the liquor entering the second effect must be heated to 180°F, and that entering the first effect to about 250°F. The heat required to raise the temperature of the liquor, plus the heat lost by radiation from the surface of the equipment, reduces the amount of water evaporated by 1 lb steam to about 2.2 lb.

The heart of the evaporator system is a group of external heaters. Heat must be quickly and efficiently transferred from the condensing steam to the liquor or the process will slow down. The heaters are composed of a vapor space into which the steam

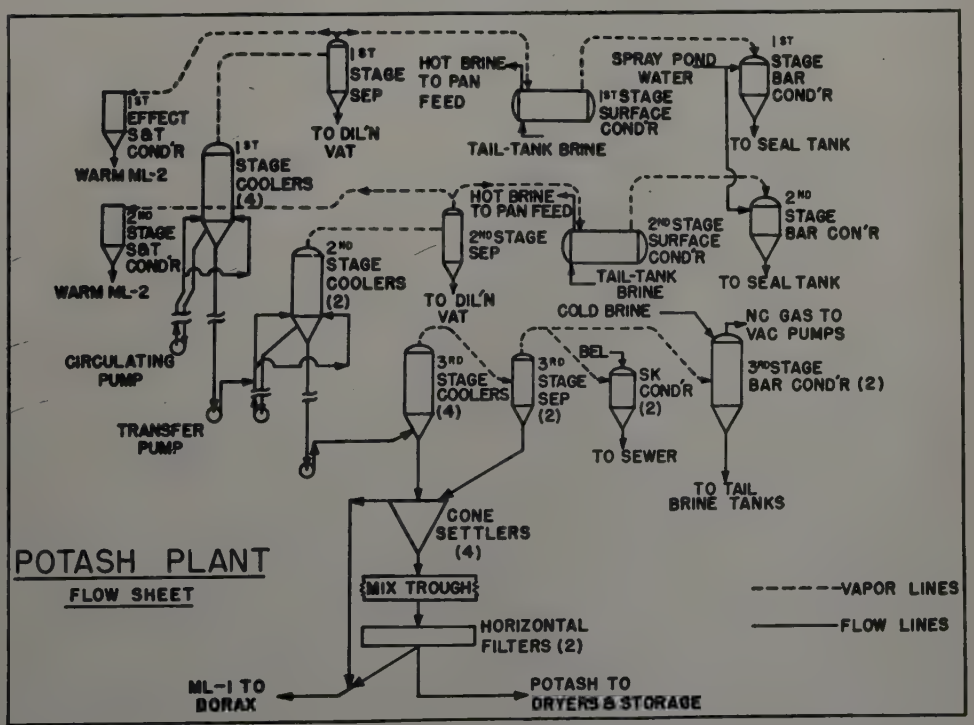
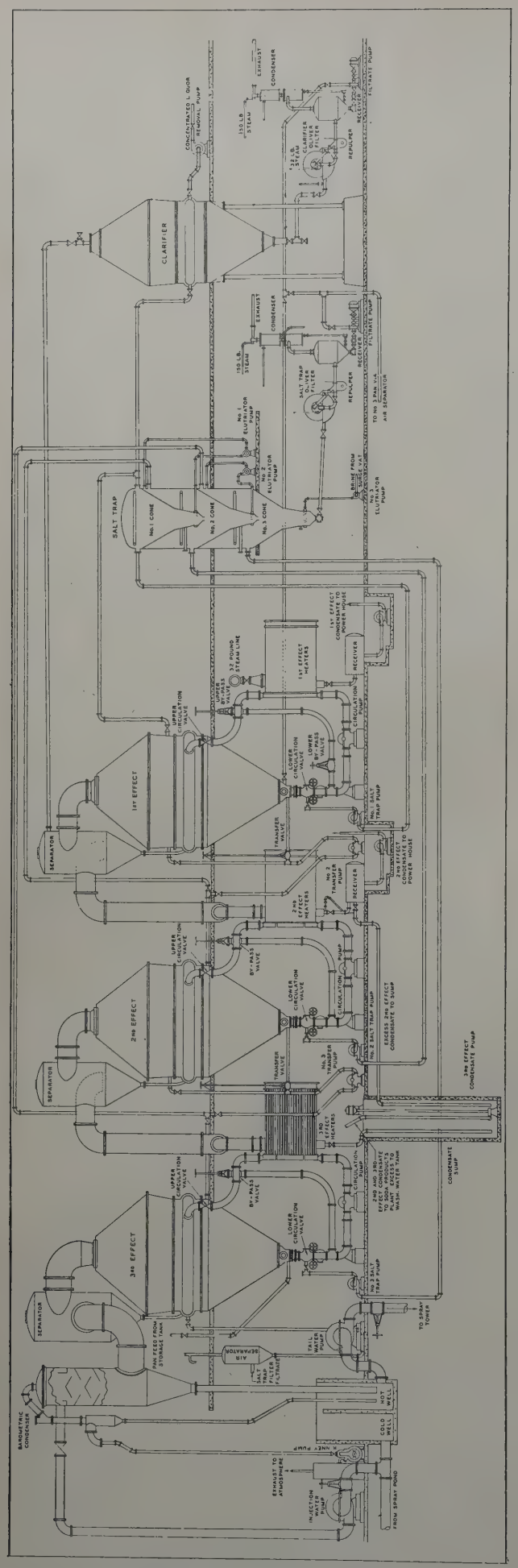


Fig. 3—In the crystallization process, the second step of the main plant cycle, the concentrated liquor is cooled to 100°F, the crystallized potash is settled in settling cones, and the settled material is filtered from the liquor in two 15-ft diam horizontal Oliver filters.



or vapor is introduced. Across the vapor space are stretched a number of tubes which carry the liquor back and forth four times in its passage through the heater. Condensate and noncondensable gas outlets are provided for the vapor space of the heaters. Heat passes from the steam condensing on the tube surfaces to the liquor within the tubes. The rate at which this transfer takes place depends on the following factors.

1—Steel tubes are used in the first-effect heaters and lead-coated Admiralty metal tubes in the second and third effect heaters. Steel tubes are satisfactory in the first effect because the virtual absence of oxygen from the powerhouse steam eliminates the corrosion problem. Aeration of steam from the first and second effect evaporators makes the use of lead-coated tubes necessary in the second and third effect heaters to prevent formation of a rust scale which would retard heat transfer. The lead coating also promotes dropwise rather than filmwise condensation which is desirable for better heat transfer. The scouring action of the salt prevents the formation of rust on the interior of the tubes.

2—The heater tube area was calculated to use up all the 35 psi steam produced by the powerhouse and refrigeration engines.

3—Heat transfer is effected by the difference in temperature between the liquor in the tube and the steam condensing on the outer surface of the tubes. Since the temperature of the liquor is rising as it travels through the heater, the heat transfer depends on the average temperature difference. The most accurate figure is the log mean temperature difference:

$$LMTD = \frac{T_{L_2} - T_{L_1}}{\text{Log}_e \dfrac{T_S - T_{L_1}}{T_S - T_{L_2}}}$$

This method takes into account the change in temperature of the liquor as it travels through the heater tubes.

4—The low pressure steam to the first effect evaporator heaters is held at 35 psi. A change of 2 lb in steam pressure would give about 2°F temperature change, whereas a change of 0.1 in. mercury pressure in the third-effect evaporator pan would have a 2°F temperature change. For this reason it is important to maintain minimum pressure on the third effect pans. The vacuum on this system is maintained by circulating large volumes of water over the barometric condensers to condense third effect vapors. Two Kinney pumps per unit are used to remove the non-condensable gases formed during evaporation.

5—The liquor being a poor conductor of heat, it is desirable to have all particles of liquor come in contact with the tube surface to obtain maximum heat transfer to the liquor. This condition is maintained by circulating liquor through the tubes at a velocity of 4 to 5 fps.

6—The tubes are of 2-in. diam to give maximum surface and allow enough area to prevent plugging with salt.

Sodium chloride, burkeite, sodium carbonate monohydrate, and lithium-sodium phosphate are continually crystallizing from the liquors in the pans, and on the surface of the heater tubes. It is necessary that the heaters and the evaporator units be washed out on schedule to maintain the efficiency

Fig. 4—Diagram at left indicates operating data of evaporator units, salt traps, and interconnected auxiliaries.

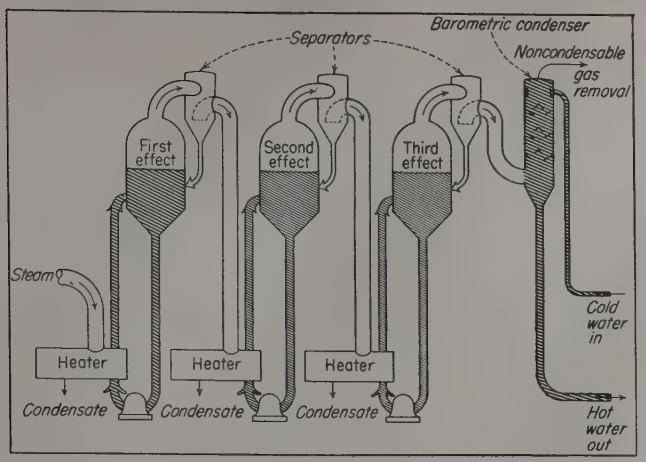


Fig. 5—Liquor advancement through triple-effect evaporators.

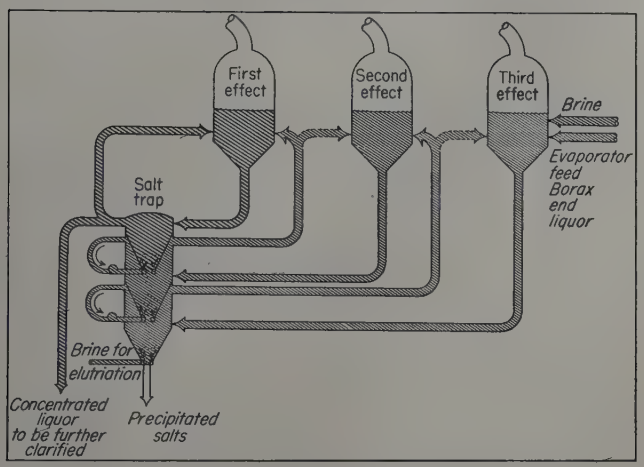


Fig. 6—The triple-effect evaporator unit heating arrangement.

of the evaporator units. The lithium sodium phosphate deposits in the first effect heater tubes, necessitating periodic acid washes to remove this scale.

In addition to salting, the evaporators have trouble with foaming and liquor entrainment in the vapors. Both cause losses from the concentration system. Droplets of liquor, thrown above the liquor surface by violent boiling, are carried upward and out of the evaporator by streams of vapor. These droplets are caught in the vapor separators and returned to their respective pans, see Fig. 6.

Foam caused much trouble at first, when these evaporators were developed. After much study, a cheap mixture of fatty acid, containing lauric acid, is continually added. This weakens the surface film and eliminates the foaming trouble.

In the evaporators some carbon dioxide is driven out of the brine by a reaction which may be expressed in the following manner:

$$Na_2B_4O_7 + Na_2CO_3 - 2Na_2B_2O_4 + CO_2$$

Owing to increased alkalinity, borate in the plant liquors becomes more soluble. This reaction is reversed by the addition of the carbonation plant borax which contains 20 to 30 pct $NaHCO_3$.

$$2Na_2B_2O_4 + 2NaHCO_3 - Na_2B_4O_7 + 2Na_2CO_3 + H_2O$$

The second step of the main plant cycle is the crystallization process. Here the concentrated liquor is cooled to 100°F and a crop of potash is recovered, see Fig. 6.

By checking it is obvious that concentrated liquor contains more KCl and $Na_2B_4O_7$ than pan feed, and contains less NaCl, Na_2SO_4, Na_2CO_3, and H_2O than pan feed. This is the result of evaporating H_2O and precipitating and removing the above salts from solution in the evaporating units.

The potash crystallization process is composed of cooling, settling, and filtering operations. This is accomplished by cooling the hot concentrated liquor to 100°F in vacuum coolers, settling the crystallized potash in settling cones, and filtering the settled material from the liquor in two 15-ft diam horizontal Oliver filters.

It is important to have a concentrated liquor almost saturated with respect to KCl. Since the liquor is saturated with NaCl, if KCl concentration is low there must be extra dilution to keep NaCl from crystallizing during KCl crystallization.

Table I. Analysis of Concentrated Liquor

Item	KCl, Pct	$Na_2B_4O_7$, Pct
Pan feed	7.5	3.8
Concentrated liquor	19.5	10.0
Mother liquor No. 1	11.5	9.8
Mother liquor No. 2	11.4	7.5
Brine	5.0	1.6

For example, if the liquor contains 37 g KCl per 100 g of water it would require only enough addition of H_2O to replace the amount lost from the liquor by evaporation in the potash vacuum coolers. On the other hand, a low-gram liquor (34 g per 100 H_2O) would require extra dilution to keep the sodium chloride in solution during crystallization in the vacuum coolers, thereby reducing the yield of potash and borax per cycle. The extra water must be evaporated in the concentration cycle.

In the diagram, Fig. 7, two polytherms are shown: one is the solubility curve for KCl in the temperature range of the KCl crystallization process; the other is the solubility curve for borax through the same range. Points A and A′ represent the percentage of KCl and $Na_2B_4O_7$ in the concentrated liquor. Points B and B′ represent the precentage of these two substances after dilution water has been added. Points D and D′, E and E′, and F and F′ represent the percentage of KCl and borax respectively in the liquors leaving the first, second, and third stages of cooling. Point C represents the approximate tem-

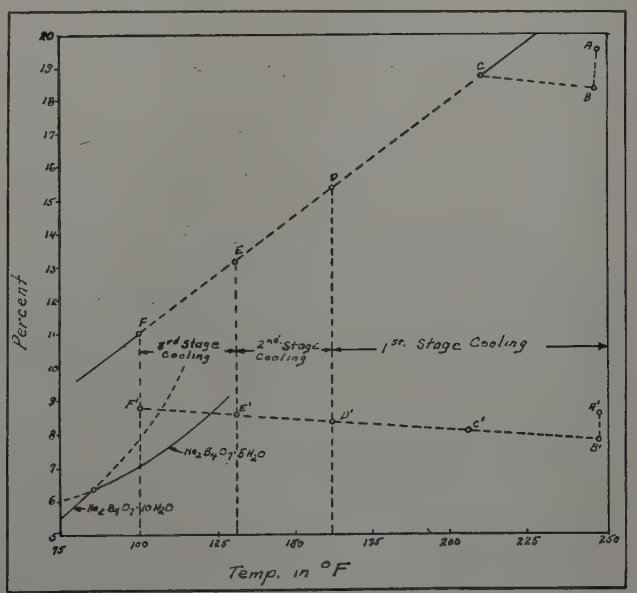


Fig. 7—Solubility curves for KCl and borax—see text.

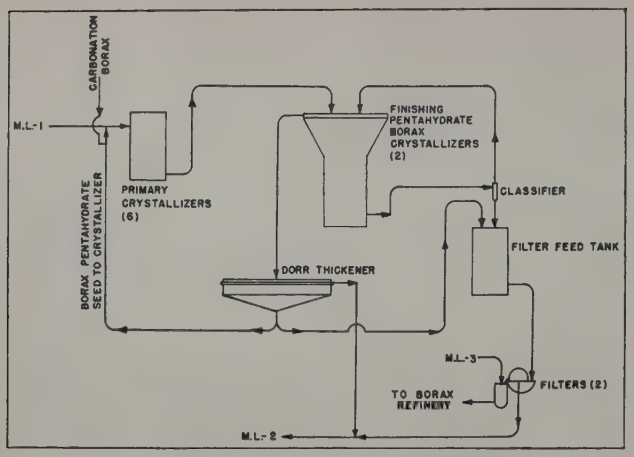


Fig. 8—Pentahydrate borax plant.

perature at which the diluted concentrated liquor becomes saturated with respect to KCl.

The line B′ E′ represents the path of borax concentration during the cooling process. Note that the concentration of borax remains almost the same between B′ C′ and increases slightly between point C′ F′. The reason for this increase in concentration is that water is evaporated and potash is removed, causing a reduction in the total weight of the liquor. Note also that the line C′ F′ passes through the borax solubility curve instead of following down the curve to lower concentrations as in the case of potash (KCl). It is evident that potash begins to crystallize from the liquor at about 205°F and continues to crystallize throughout the cooling range, while borax remains in solution. Furthermore, it is evident that the liquor becomes supersaturated with respect to borax while passing through the final stages of the KCl crystallization process. The reasons for the supersaturation are that in the tetraborate pentahydrate field there is an extremely slow rate of nucleus formation. The linear rate of pentahydrate crystal growth is also slow in these liquors.

The tendency for a hydrated salt-like borax to crystallize as a metastable solid phase at a temperature above its stable range is much more than at temperatures below its transition point. Thus because of high rate of liquor flows through the third-stage potash coolers, short retention time, and lack of violent agitation, the supersaturation conditions are easily maintained.

In vacuum cooling heat is removed by the vapors in the vacuum crystallizer; thus the liquor is cooled. The reasons for cooling this liquor in three stages instead of one are as follows: more efficient use of the coolant, better control of the borax impurities, and smaller vapor lines from coolers to condensers, see Table II.

Note that volume of vapors from the third stage is approximately five times that of the first stage.

Raw brine is used as a cooling medium instead of H_2O for two reasons: 1—Brine coming from the lake at 76°F is cooler than any available cooling H_2O during the summer months. 2—Brine has a lower vapor pressure than H_2O and thus is equivalent to H_2O of 64°F when used in a barometric-type condenser.

The brine is used through two barometric condensers on the third stage and then pumped in series through two tube condensers: first through the second stage, then through the first stage, thus recovering a maximum amount of heat from liquors being cooled. The brine is heated to 125°F while cooling the liquor to 100°F. In recent years, owing to increased capacity of the concentration process, it has been necessary to supplement the third-stage cooling with 400 gpm of soda products plant end liquor (BEL) which is sent to the coolers at 72°F.

The potash end liquor (known as ML1) at 100°F is pumped to the borax plant. This liquor is saturated with potash and supersaturated with respect to sodium tetraborate pentahydrate. This liquor is seeded with fine sodium tetraborate pentahydrate from the borax plant Dorr thickener underflow and carbonation plant borax and sodium bicarbonate. This liquor is fed into two parallel sets of three primary crystallizers connected in series. The overflow from the primary crystallizer is pumped into two large agitated crystallizers operated in parallel. These tanks have approximately 45 min retention time and maintain sludge density of 40 to 44 pct sodium tetraborate pentahydrate as a seed mixture.

In this manner the stable sodium tetraborate pentahydrate is brought to equilibrium in solution. The coarse crystals are removed from solution by circulating the sludge from the bottom of the crystallizer through two cyclone classifiers. The coarse crystals are discharged from the bottom of the classifiers into a filter feed tank, whereas the fines are returned, as seed, to the system with the solution out of the top of the cone separators, see Fig. 8.

Table II. Volume of Vapors

Stage	Vapor Temp.	Liquor Temp.	Specific Volume of H_2O Vapor, Cu Ft Per Lb
3rd stage	80°F	100°F	632.9
2nd stage	110°F	130°F	265.4
1st stage	140°F	160°F	123.0

The overflow from the main crystallizers contains 8 to 15 pct fine suspended solids. This liquor flows to a 60-ft Dorr thickener. The overflow from the thickener, ML2, and the filtrate from the crude Oliver filters is combined and pumped through the Potash plant where it is used as a coolant in the first stage vertical tube condensers, then pumped to pan feed tanks where it is mixed with hot brine to form feed for the evaporator units. The underflow from the thickener is pumped back to the primary crystallizers as seed, see Fig. 8.

The crude tetraborate pentahydrate can be processed crude by pumping it directly to the Sharples centrifugals and drying it (this is sold as Tronabor), or it can be pumped on to two 13-ft diam horizontal Oliver filters, dewatered, washed, redissolved, filtered to remove physical impurities, and either recrystallized as refined tetraborate pentahydrate or decahydrate, depending on market requirements for these products.

Acknowledgments

This paper was condensed from company research department files and from W. A. Gale's report on the geology of Searles Lake presented at the AIME meeting in Los Angeles, February 1953. This process and equipment has been modified, developed, and adapted to process Searles Lake brine by the many engineers who have worked in the research and production organization since 1916, when C. P. Grimwood built the first evaporator unit. P. S. Dunn, Vice President in charge of technical operations, and R. W. Mumford, Vice President and Consultant Engineer for American Potash & Chemical Corp., made it possible for the writer to present this paper and helped plan and organize the potash symposium presented at the AIME meeting in 1953.

Flotation Characteristics of Pyrrhotite with Xanthates

by C. S. Chang, Strathmore R. B. Cooke, and Iwao Iwasaki

The effects of aeration on an aqueous suspension of pyrrhotite were studied and their results correlated with flotation tests using xanthates as collectors. The effects of copper activation and of pH variation were determined and possible mechanisms postulated.

PYRRHOTITE has long been considered a gangue mineral to be eliminated as tailing in the treatment of various sulphide ores. However, in recent years the world-wide lack of sulphur resources has called attention to this mineral as a potential source of both sulphur and iron. Its importance as an economic mineral, however, has not been particularly emphasized. For this reason very little is known about its response to flotation, except that it can be depressed easily in alkaline circuit, by long aeration,[1, 2] addition of oxidizing agents,[3] or by starch.[4]

The object of this work was to study the floatability of pyrrhotite. This includes the effect of oxidation by aeration, of copper activation, and of change in pH.

Preparation of the Pyrrhotite Sample: It was desirable that the highest grade of pyrrhotite obtainable be used for this experiment, since the presence of other minerals could affect the surface properties.[5] However, no pyrrhotite was available as crystals, and massive deposits of hydrothermal origin commonly contain considerable amounts of chalcopyrite. Pyrrhotite concentrate was, therefore, prepared from a sulphide deposit occurring near Aitkin, Minn. The deposit is of pyrometamorphic nature consisting mainly of pyrrhotite and pyrite with graphite, silicates, and carbonates as gangue.

The ore, already crushed through 3 mesh when received, was screened at 65 mesh and the undersize discarded. The oversize was crushed through rolls, and then stage-ground dry in an Abbe porcelain mill, the −65 mesh portion being screened out after every 15 min of grinding until all the material passed through this size. The ground product was then concentrated with a drum-type dry magnetic separator. The rougher concentrate was cleaned twice and then demagnetized. The final product was split in a Jones splitter and stored in air-tight bottles. Microscopic examination of the concentrate showed that it was relatively clean and free of pyrite, locked particles, and gangue. By means of the krypton gas adsorption method,[6] the specific surface was determined to be 3000 cm^2 per g. The chemical and screen analyses of the final concentrate are given in Tables I and II respectively.

It is a well-recognized fact that the oxidation of some sulphide ores during stockpiling, grinding, and conditioning affects their flotation behavior. The problem of oxidation may become serious in the case of pyrrhotite, since this is known to be more easily oxidized than many other sulphides. To ascertain the extent of oxidation, an experiment was carried out by aerating an aqueous suspension of pyrrhotite with air, oxygen, and nitrogen as follows.

A 300-g sample of pyrrhotite in 2700 ml of water was agitated and simultaneously aerated in a Fagergren-type laboratory flotation machine. A Precision wet test meter was connected to the air inlet valve, the flow rate of the gas being kept constant at 0.3 cu ft per min throughout the experiment. Samples of approximately 30 ml each were taken from the cell at 0, 4, 10, 20, 35, 60, and 90 min. After the pH was taken, each sample was filtered and the filtrate was analyzed for total iron and sulphur.

The iron was determined colorimetrically by the thioglycolate method using a green filter.[7] The filtrate was oxidized with bromine to convert all of the soluble sulphur compounds into sulphate and this was determined with a Parr turbidimeter.[8]

When aeration tests were made in alkaline circuit, calcium hydroxide or sodium hydroxide was added at regular intervals to maintain a constant pH.

A similar procedure was followed in an experiment to determine the abstraction of copper ion by pyrrhotite. In this case various quantities of cupric chloride were added. The filtrate from each sample taken was analyzed for copper, total iron, and sulphur. The carbamate method with a green filter was used for the copper analysis,[7] since this method could tolerate a considerable amount of iron in the solution.

A pneumatic cell, made from a 350-ml fritted glass Buechner funnel, was used for this experiment. The detail of the assemblage has been described elsewhere.[9] In the present work a stainless steel baffle was inserted in the cell. This baffle overcame the tendency for the coarse pyrrhotite particles to be swirled around the wall of the cell and thus fail to collect in the froth.

A 50-g sample of pyrrhotite was added to the cell which contained 260 ml of water. When pretreatment of the sample was desired, reagents, such as activator and pH regulator, were then added and the pulp was conditioned for a specified conditioning time. Prior to the addition of the collector approximately 15 ml of the solution were removed for pH measurement and for iron and sulphur analyses. Copper when used as activator was also determined. Collector and frother were then added and the pulp was conditioned for an additional 2 min. Air was admitted to the cell and the froth removed. The separation required from 4 to 6 min, depending on the characteristics of the froth.

The float and non-float products were filtered, dried, weighed, and assayed for iron.

C. S. CHANG, Junior Member AIME, S. R. B. COOKE, Member AIME, and I. IWASAKI, Student Associate AIME, are respectively, Research Associate, Professor of Metallurgy and Mineral Dressing, and Teaching Assistant, School of Mines and Metallurgy, University of Minnesota, Minneapolis.

Discussion on this paper, TP 3680B, may be sent (2 copies) to AIME before March 31, 1954. Manuscript, June 11, 1953. New York Meeting, February 1954.

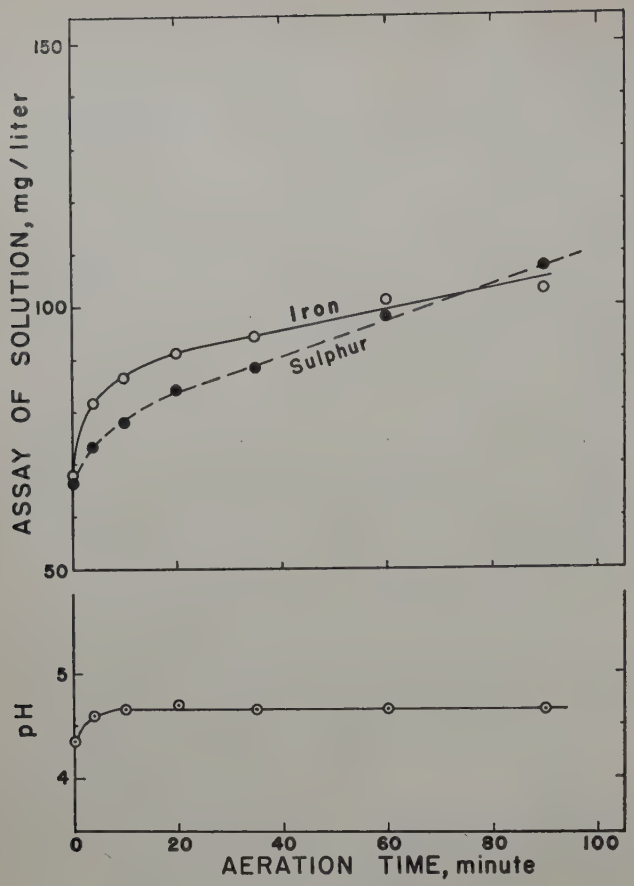


Fig. 1—Aeration test with air.

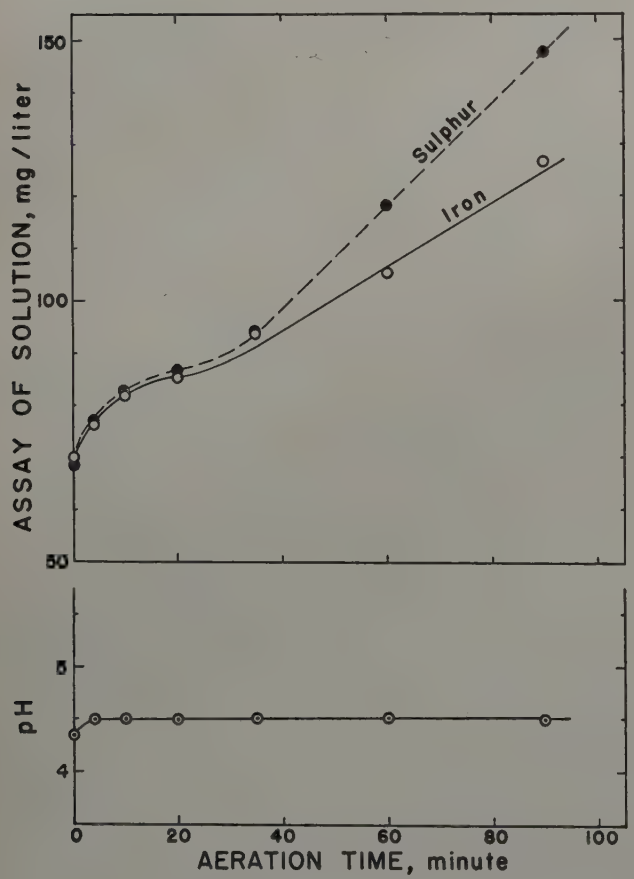


Fig. 2—Aeration test with oxygen.

After each flotation test, the cell was thoroughly washed with hot water and rinsed with demineralized water. Occasionally the cell was boiled with aqua-regia to remove fine mineral particles plugging the pores of the fritted glass.

Table I. Chemical Analysis of Pyrrhotite Concentrate

Item	Pct
Fe	48.21
S	31.18
SiO_2	12.74
Al_2O_3	2.73
Total C	1.21
Graphite C	0.88

Table II. Screen Analysis of Pyrrhotite Concentrate

Size, Mesh	Wt, Pct	Fe, Pct
−65 +100	17.64	55.76
−100+150	14.96	55.92
−150+200	11.95	56.08
−200+270	6.20	54.47
−270+400	12.28	47.65
−400	36.97	38.09
	100.00	48.21

The following reagents were used for the preceding tests:

Collector: The potassium ethyl and n-amyl xanthates used in this experiment were synthesized in the usual way.[10] All the chemicals used for these preparations were of analytical reagent grade. The products obtained were placed in evacuated pyrex bottles and stored in a refrigerator. In general, a 1.0 pct solution was used for addition.

Frother: Several frothers were tested and B-23 gave the best frothing characteristics. It produced a fairly dry froth consisting of medium-sized bubbles, and a comparatively small quantity of material was mechanically carried over. The frother was diluted 20 times by volume with ethyl alcohol and 1 to 2 drops of the solution were usually added. One drop of this solution was equivalent to 0.02 lb per ton of ore.

Other Reagents: The pH regulators, such as sodium hydroxide, hydrochloric acid, calcium oxide, and sulphuric acid, and the activator, cupric chloride ($CuCl_2 \cdot 2H_2O$), were of analytical reagent grade. The nitrogen and oxygen used had a listed purity of 99.5 pct. Demineralized water containing less than 0.1 ppm of salts as NaCl was used in the preparation of all solutions and for all test work.

Experimental Results

Aeration and Copper-Abstraction: A preliminary test was made for the presence of ferrous, ferric, and sulphate ions in the filtrate from an aerated aqueous suspension of pyrrhotite. All the iron present was in the ferrous state and remained so for a period which was of the order of several days to a few weeks. Sulphate ion was also present, but the quantity increased when the filtrate was oxidized with bromine. This indicated the presence of soluble sulphur compounds other than sulphate.

1—Aeration at Natural pH: Three aeration tests were made with air, oxygen, and nitrogen respectively. These three tests were performed within one week to minimize any serious sample alteration during storage. The results are plotted against time in Figs. 1, 2, and 3. They show that liberation of soluble ions is rapid in early stages but approaches a constant rate as aeration time is increased.

With nitrogen, Fig. 3, initial liberations of iron and sulphur are the highest of all. After about 20 min of aeration, however, both iron and sulphur pass into solution at the same rate. With oxygen, Fig. 2, and after the initial stage, the rate of liberation of soluble ions is greater than with air, Fig. 1. In both cases the rates for sulphur are greater than those for iron, suggesting precipitation of the iron during the process. The samples from these tests, when dried, gave a much more reddish color than those obtained with nitrogen.

Constant pH values of 4.5, 4.65, and 5.1 were reached with oxygen, air, and nitrogen respectively and will henceforth be referred to as the corresponding *equilibrium pH*. This would seem to indicate that the pyrrhotite provides buffering action to the pulp.

2—Aeration at Alkaline pH: A similar experiment was carried out with air in an alkaline circuit. The pH was maintained at approximately 8.5 by the addition of calcium hydroxide or sodium hydroxide at regular intervals. Results are shown in Fig. 4.

No iron was present in the solution. The sulphur was liberated in the same manner as in the previous experiment but in larger quantity and at a higher rate. At equivalent pH, the rate was greater for sodium hydroxide than for calcium hydroxide.

3—Aeration in Presence of Cupric Ion: Aeration tests with addition of 0.7, 3.4, and 6.9 lb of cupric chloride ($CuCl_2 \cdot 2H_2O$) per ton were made. These represented initial concentrations of 14.4, 71.7, and 143.5 mg respectively of copper per liter of solution. Only air was used in this experiment. The results are given in Figs. 5, 6, and 7. They show in all cases a depletion of copper from solution, in agreement with the early finding of McLachlan.[11] The time for complete copper removal depends on the initial copper concentration. The amount of sulphur passing into solution is almost constant regardless of the amount of copper added, and it is relatively less than that when copper is absent. However, the quantity of iron liberated depends on the quantity of copper abstracted.

It should be noted that the initial sample was taken after 15 sec of agitation without aeration and a considerable amount of copper was removed from solution during that time. This accounts for the lower copper concentrations reported at zero time compared with that initially added.

To investigate the possibility of any color change due to activation, polished specimens of massive pyrrhotite from Kimberley, B.C., mounted in lucite, were agitated in 0.35 and 1 pct cupric chloride solutions for 15 hr at room temperature, 25°C. One half of each specimen was coated with paraffin while the other half was exposed to the copper solution. For photomicrographs of tested specimens, see Fig. 8.

Depending on the time of exposure to the cupric chloride solution the exposed surfaces ranged from a brass-yellow to a highly-irridescent peacock color. Local precipitation of an unknown substance roughened the surface. The specimens contained inclusions of chalcopyrite, sphalerite, and pyrite, and these showed no change in color. Some difference in shade of color was noted among the pyrrhotite grains, Fig. 8b, indicating that copper attacked preferentially along certain crystallographic directions.

A similar experiment was made in a 10 pct cupric chloride solution at a temperature of 50°C. The pyrrhotite surface was completely obscured by a

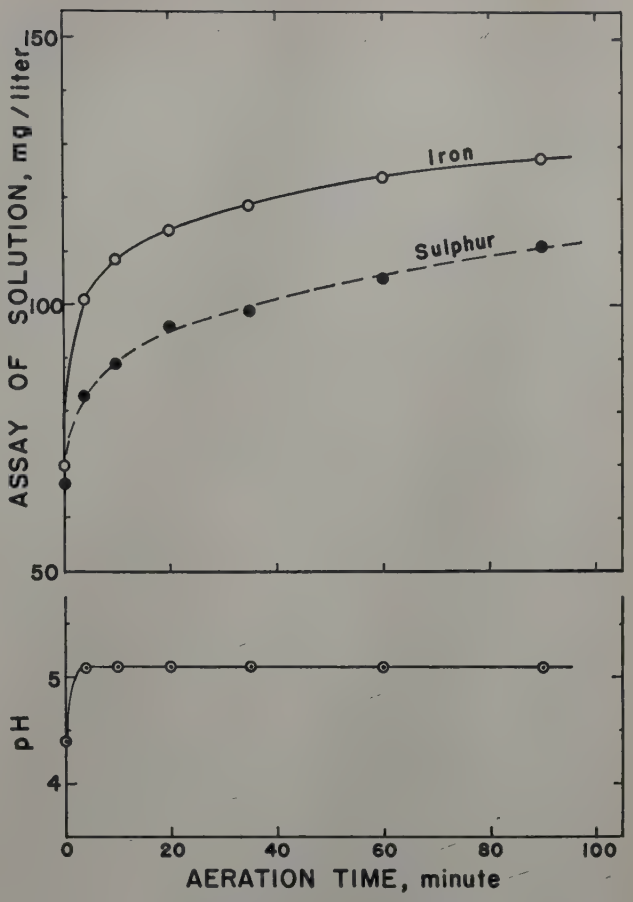


Fig. 3—Aeration test with nitrogen.

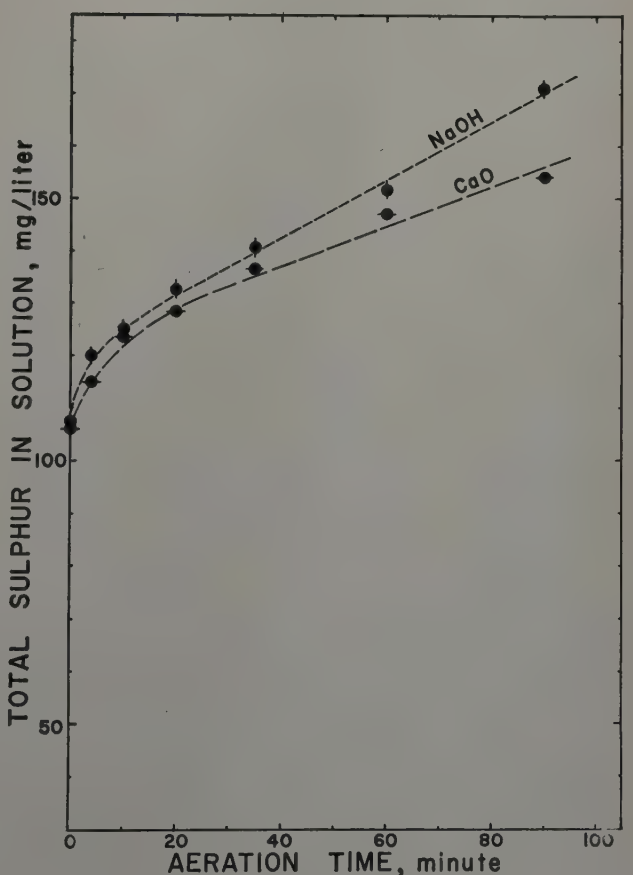


Fig. 4—Aeration test at pH = 8.5.

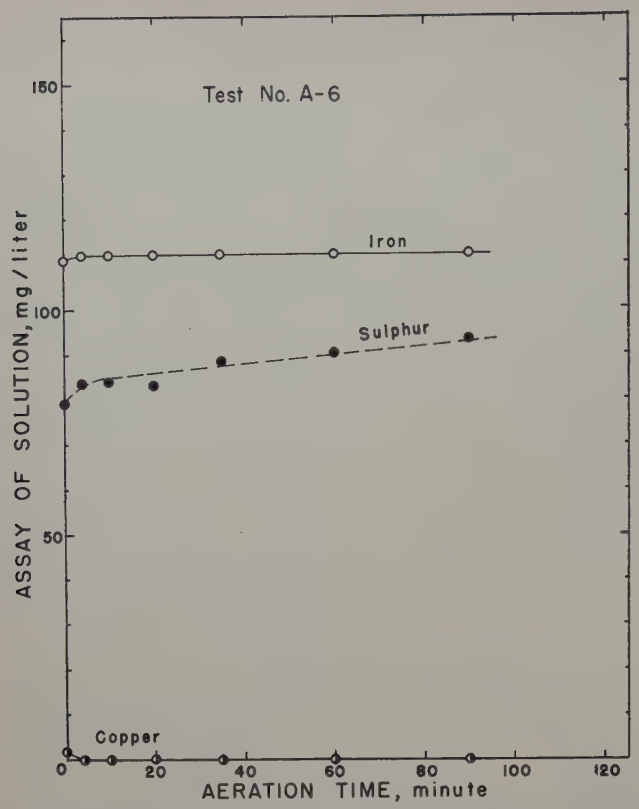


Fig. 5—Aeration test with 0.7 lb $CuCl_2 \cdot 2H_2O$ per ton.

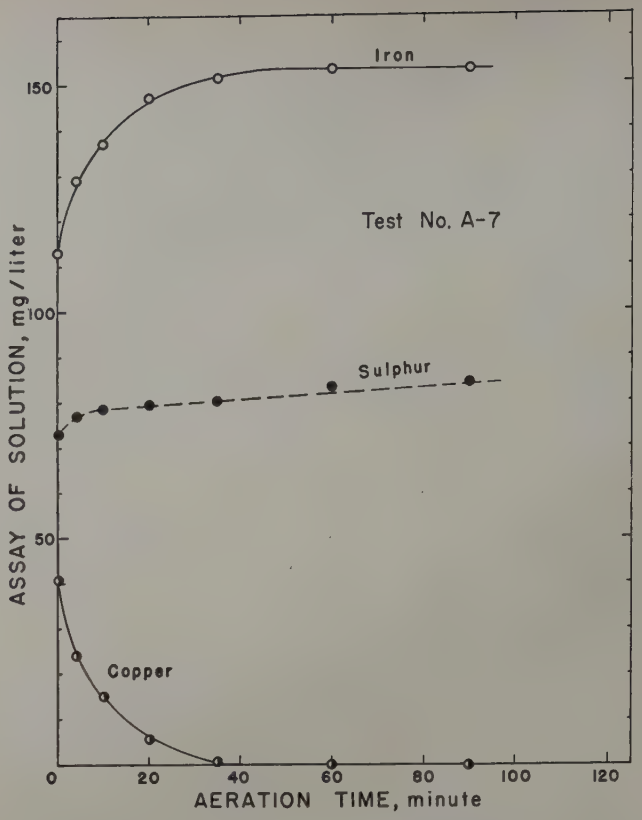


Fig. 6—Aeration test with 3.4 lb $CuCl_2 \cdot 2H_2O$ per ton.

precipitate containing iron but otherwise of unknown composition.

Flotation: All flotation tests, unless otherwise specified, were made at the natural pH of the pulp, i.e., 4.5.

1—Effect of Collector Addition With and Without Copper Activation: Two series of flotation tests were performed. Potassium ethyl xanthate was used as collector in one series and potassium amyl xanthate in the other. The collector addition ranged from 0.4 to 6.4 lb per ton of pyrrhotite. In two similar series of tests pyrrhotite was first conditioned with 1 lb cupric chloride per ton for 15 min prior to flotation.

All the data from these flotation tests are plotted in Fig. 9. Without copper activation the recovery was rather poor and even at a collector addition of 6.4 lb per ton, the recovery was only 28.9 pct with ethyl xanthate and 53.3 pct with amyl xanthate. On the other hand, with prior activation, the recovery was increased approximately 20 pct at the same collector addition. However, the general shape of the curves indicates that it would require extremely large quantities of collector for complete recovery. It was noted from visual observation that non-float products consisted mainly of the coarser portion of the original sample.

2—Effect of Collector Addition in Stages: In this experiment, the collector was added in stages and float products were collected separately after each addition. The cumulative amount of collector at each stage was arranged to correspond with those of single addition in the previous experiment. The results are shown in Fig. 10 with the cumulative percent iron recovered plotted against cumulative amount of collector added. Comparing Figs. 9 and 10, it can be seen that the same total amount of collector, added stagewise, gives much improved recovery. For example, 3.2 lb amyl xanthate per ton added in stages gave about 96 pct recovery of activated pyrrhotite, whereas a single addition of the same quantity of collector gave a recovery of only about 50 pct.

To study the size distribution, screen analyses were made on all flotation products obtained in these tests. The cumulative percent weight recovered in each size fraction is plotted in Figs. 11 and 12 against the cumulative amount of collector added.

The results show that the floatability of pyrrhotite decreases with increase in particle size. This tendency is most pronounced when ethyl xanthate is used as collector to float unactivated pyrrhotite with the +200 mesh particles responding very reluctantly to flotation. The results also confirm the increase in floatability with copper activation.

3—Detrimental Effect of Excess Cu^{++} ion in Pulp: Even though copper activation of pyrrhotite increases its floatability, the presence of excess Cu^{++} ion in the pulp precipitates xanthate according to the following reaction, and the presence of excess Cu^{++} ion is undesirable.

$$4\,ROCSS^- + 2\,Cu^{++} \rightarrow 2\,ROCSS \cdot Cu + (ROCSS\text{-}SSCOR)$$

This is demonstrated by two flotation tests in which the pyrrhotite was conditioned with 5 lb of cupric chloride per ton for 15 min and 2 hr respectively and the amyl xanthate was added in increments. The results are given in Fig. 13 together with those of Test No. 22 already shown on Fig. 10. Copper analyses were made on the pulp solution prior to collector addition and the results are given in Table III.

Fig. 13 shows that for the same recovery more collector is required as excess Cu^{++} ion is increased in the pulp. With Test 23 the recovery rises slowly at first, then suddenly increases toward completion. The amount of potassium amyl xanthate necessary

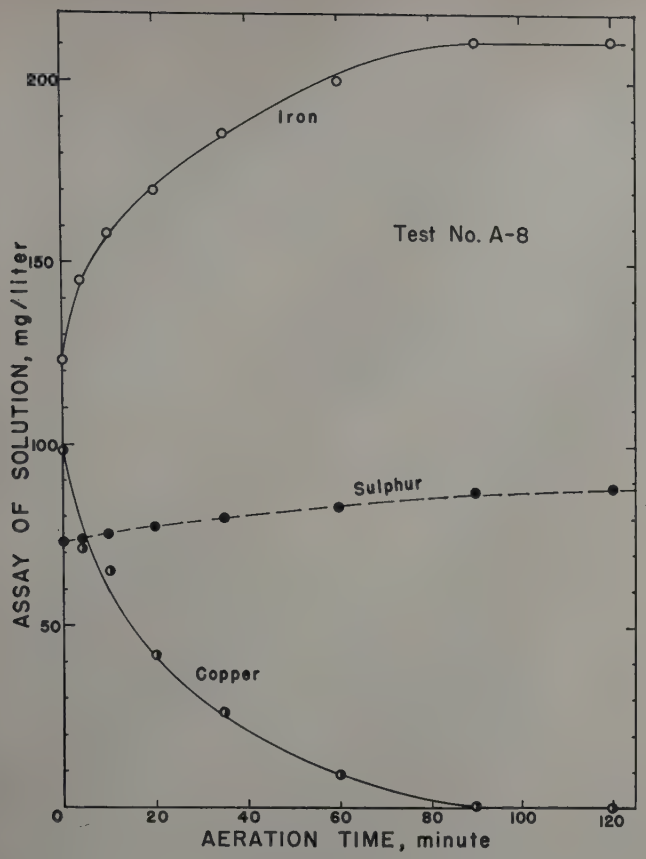


Fig. 7—Aeration test with 6.9 lb $CuCl_2 \cdot 2H_2O$ per ton.

to precipitate Cu^{++} ion roughly corresponds to the projected intersection of the two parts of the curve.

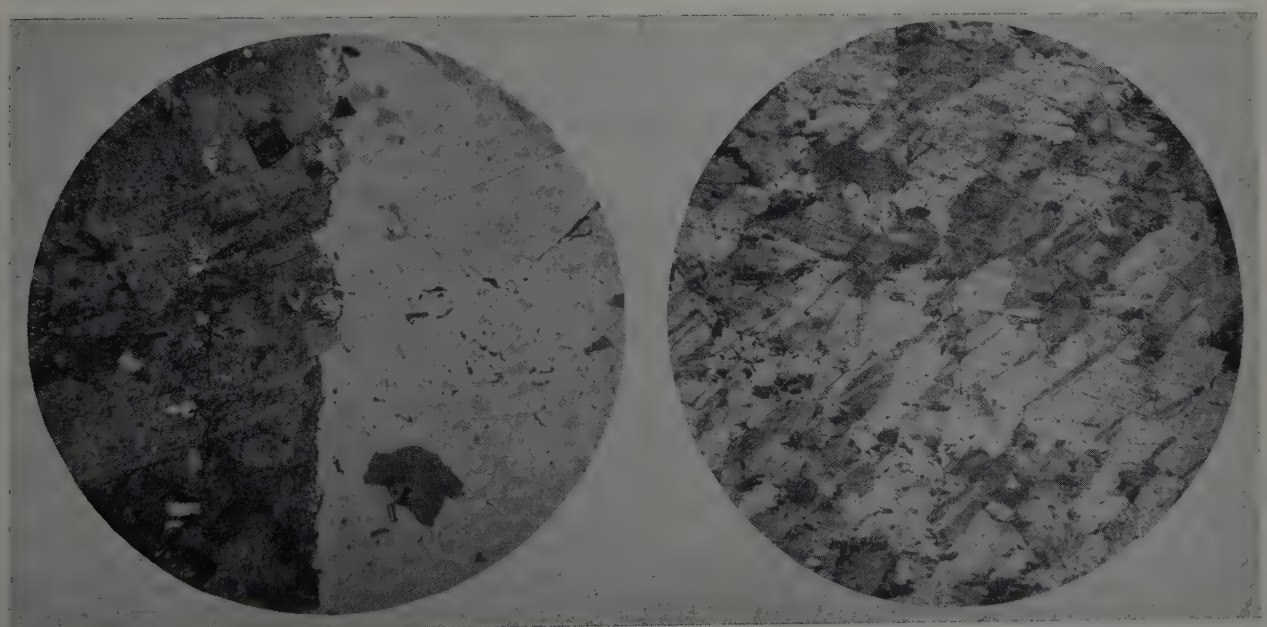
In a separate test the equivalent of 6.4 lb of amyl xanthate per ton was mixed in the cell with a stoichiometric excess of Cu^{++} ion (equivalent to 3.5 lb of cupric chloride per ton) prior to the addition of pyrrhotite. Flotation with this mixture as a collector gave a recovery of approximately 18 pct, presumably due to the independent collecting ability of cuprous xanthate and dixanthogen, and may account for that flotation which takes place prior to the first inflection point.

4—Effect of pH on Unactivated and Activated Flotation: A preliminary flotation experiment at a pH of approximately 2.5 with amyl xanthate indicated that 1 lb of this collector per ton was sufficient to give almost complete recovery. One series of tests was made at this collector addition, and the pH was varied from 2 to 11 by adding hydrochloric acid, sulphuric acid, sodium hydroxide, or calcium hydroxide. Another series was run at an amyl xanthate addition of 6 lb per ton on restricted pH range.

Table III. Analysis of Cu^{++} in Pulp Solution after Activation

Item	Test 22	Test 23	Test 24
$CuCl_2 \cdot 2H_2O$ added	1 lb /ton or 25 mg	5 lb per ton or 125 mg	
Conditioning time	15 min	15 min	2 hr
Excess Cu^{++} in pulp	0.09 mg	17.2 mg	5.0 mg
Equivalent amount of amyl xanthate precipitated by excess Cu^{++}	0.02 lb per ton	4.34 lb per ton	1.25 lb per ton

The collector and the frother were added after 3 min conditioning with acid or with alkali. The pH of the pulp was measured before and after each flotation test. The average of the two values was taken as the pH during flotation. In the first series of tests total iron and sulphur analyses were made on the pulp solution prior to flotation. The results of this experiment are given in Fig. 14. In this figure a *critical pH* is sharply defined at about 4.5, which corresponds roughly to the *equilibrium pH*. Below this pH, flotation recovery is almost complete, while above this value the floatability of pyrrhotite decreases to a minimum. It was observed that at collector addition of 6 lb per ton, there was a tendency to cause over-frothing when the pH was raised beyond the *critical* value even without the benefit of a frother. This increased the amount of material mechanically carried over in the froth and accounted



Figs. 8a and 8b—Photomicrographs of copper-activated pyrrhotite. Parallel nicols, X100. Left: after conditioning in 1 pct $CuCl_2 \cdot 2H_2O$ solution for 15 hr. The part to the right was sealed with paraffin which was removed before photographing. Right: after conditioning in 0.35 pct $CuCl_2 \cdot 2H_2O$ solution for 15 hr. Note the difference in shade of color among the pyrrhotite grains.

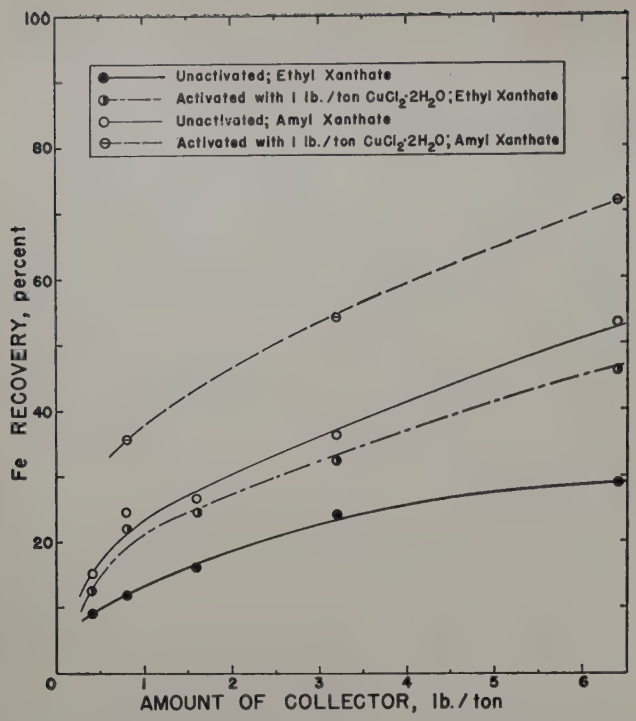

Unactivated; Ethyl Xanthate
Activated with 1 lb./ton $CuCl_2 \cdot 2H_2O$; Ethyl Xanthate
Unactivated; Amyl Xanthate
Activated with 1 lb./ton $CuCl_2 \cdot 2H_2O$; Amyl Xanthate
Fe RECOVERY, percent
AMOUNT OF COLLECTOR, lb./ton

Fig. 9—Effect of collector addition on flotation recovery.

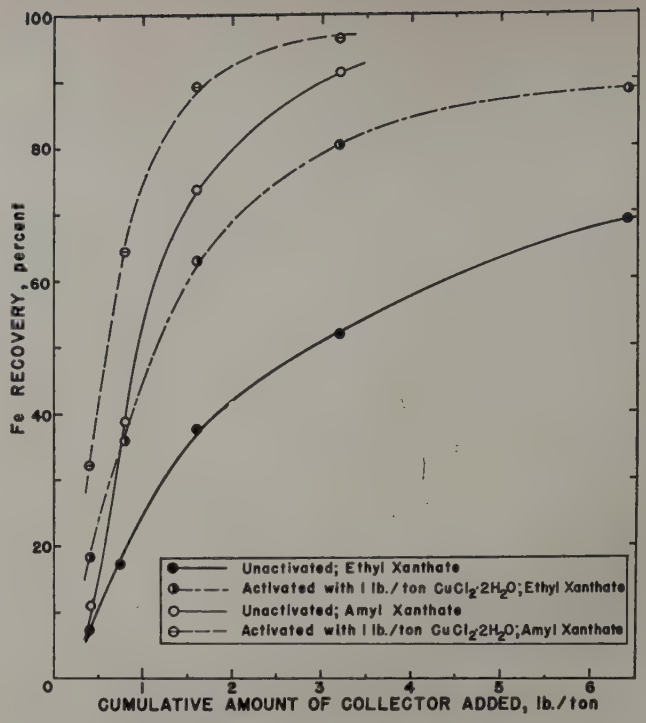

Unactivated; Ethyl Xanthate
Activated with 1 lb./ton $CuCl_2 \cdot 2H_2O$; Ethyl Xanthate
Unactivated; Amyl Xanthate
Activated with 1 lb./ton $CuCl_2 \cdot 2H_2O$; Amyl Xanthate
Fe RECOVERY, percent
CUMULATIVE AMOUNT OF COLLECTOR ADDED, lb./ton

Fig. 10—Effect of stage collector addition on recovery.

for the increase in recovery with pH in that range. After the addition of acid or alkali, however, it was also noted that the pH tended to drift toward the *equilibrium* value. For this reason a series of different recoveries was obtained at the critical pH. Taking that recovery obtained with addition of neither acid nor alkali as a reference, acid addition increased and alkali addition decreased the recovery, the increase or decrease depending on the amount of addition. In one instance sufficient acid was added to give a pH of approximately 2. Forty minutes of conditioning restored the pH to the critical and equilibrium value of 4.5, and flotation of this pulp gave the same recovery as when pH was 2, indicating that pH alone may not control floatability.

The analyses of the pulp solution show that the amount of iron increases with the addition of acid below the critical pH, Fig. 14b. The sulphur remains more or less constant throughout the same range, but hydrogen sulphide gas was evolved from

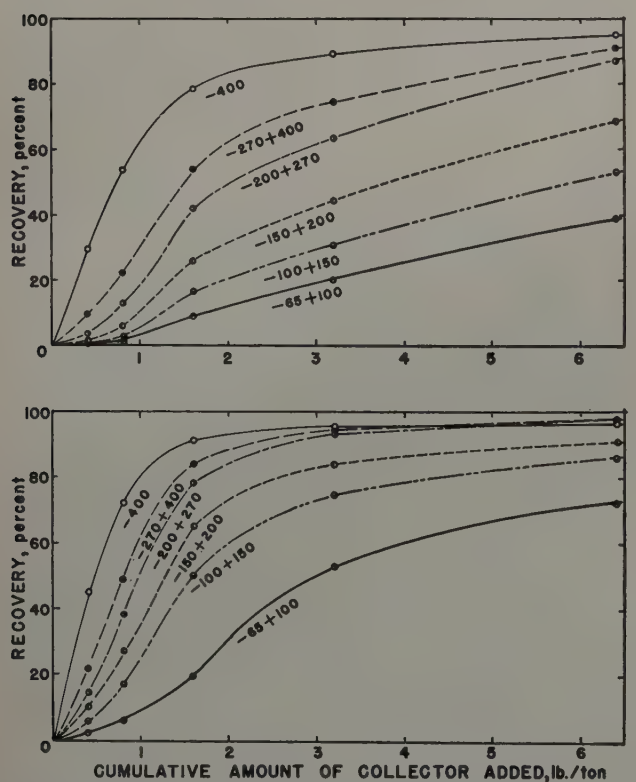


Fig. 11—Size distribution in flotation products obtained by stage addition of ethyl xanthate. Above, inactivated. Below, activated with 1 lb $CuCl_2 \cdot 2H_2O$ per ton.

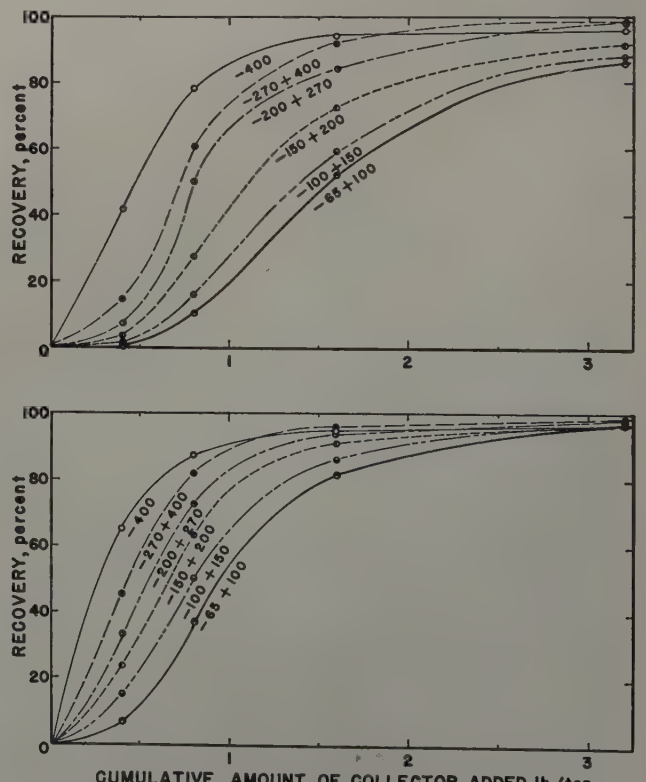


Fig. 12—Size distribution in flotation products obtained by stage addition of amyl xanthate. Above, unactivated. Below, activated with 1 lb $CuCl_2 \cdot 2H_2O$ per ton.

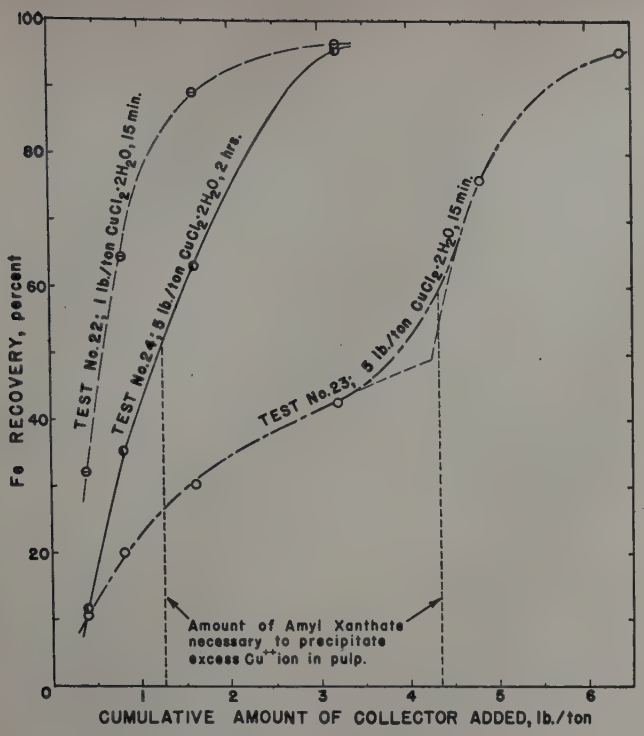

Fe RECOVERY, percent
CUMULATIVE AMOUNT OF COLLECTOR ADDED, lb./ton
TEST No.22; 1 lb./ton CuCl2·2H2O, 15 min.
TEST No.24; 5 lb./ton CuCl2·2H2O, 2 hrs.
TEST No.23; 5 lb./ton CuCl2·2H2O, 15 min.
Amount of Amyl Xanthate necessary to precipitate excess Cu++ ion in pulp.

Fig. 13—Detrimental effect of excess Cu^{++} on recovery.

the pulp. This indicates that the acid attacked the pyrrhotite and presumably cleaned the surface. Above the critical pH the amount of sulphur slightly increases with increasing pH, but the iron decreases rapidly to zero at about 7.

The effect of pH on pyrrhotite activated with 1 lb of cupric chloride per ton was also investigated. Fifteen minutes of conditioning was provided for the activation, followed by an additional 3 min of agitation with acid or alkali for pH adjustment. The results of this experiment are given in Fig. 15.

The presence of a critical pH is also indicated in Fig. 15a. The transition between flotation and depression, however, is rather gradual, ranging in pH from 3 to 5. This may be accounted for by the fact that the pH of the pulp containing copper-activated pyrrhotite drifts rather sluggishly toward the equilibrium value after the addition of acid or alkali. It was noted that a recovery of approximately 20 pct was obtained above the critical pH, compared to only 10 pct for the non-activated pyrrhotite at the same collector addition.

5—Effect of Aeration on Flotation: Flotation tests were made by first aerating different pulps with air, oxygen, and nitrogen each for 1 hr. Amyl xanthate, 1.6 lb per ton, was used as collector. The results are given in Table IV.

Table IV. Effect of Aeration on Flotation Recovery and Soluble Ions*

Test No.	Gas Used for Aeration	pH after Aeration	Analysis of Solution before Flotation: Total Fe, Mg Per Liter	Analysis of Solution before Flotation: Total S, Mg Per Liter	Fe Recovery, Pct
52	Oxygen	4.2	96	106	29.3
53	Air	4.45	110	115	48.9
54	Nitrogen	5.1	138	130	91.3

* Flotation was performed with the same gases used for aeration.

The results show that aeration with air and oxygen reduces the floatability of pyrrhotite. There is no great difference in the amount of iron and sulphur present in the three pulps.

Discussion of Results

Cupric ion is abstracted by pyrrhotite in the presence of air and its depletion from solution is proportional to the increase of iron in the pulp. The resulting activated pyrrhotite ceases to liberate iron and sulphur on further aeration. However, a considerable amount of copper is abstracted from solution in the first 15 sec of agitation. For example, in Fig. 7, 143.5 mg of copper per liter was initially added, but with 15 sec of elapsed time between addition and commencement of aeration, this had decreased in the pulp to 98.0 mg of copper per liter. Disregarding this initial loss, the amount of copper abstracted and the amount of iron liberated in Tests No. A-6, A-7, and A-8 are compared in Table V. The amount of iron in the pulp solution as given in the table is the total *less* the amount found in solution at 15 sec after adding the copper.

Table V. Relation between Copper and Iron in Pulp Solution during Aeration

Item	Test A-6	Test A-7	Test A-8
$CuCl_2 \cdot 2H_2O$ added, lb per ton	0.7	3.4	6.9
Cu abstracted, millimol	*0.05*	*1.73*	*4.13*
Fe liberated, millimol	*0.05*	*1.92*	*4.35*

The results show, within experimental error, the stoichiometric exchange reaction between copper ion and iron atoms at the pyrrhotite surface. Symmetry between the iron and copper curves (Figs. 5, 6, and 7) also suggests this exchange type of reaction.

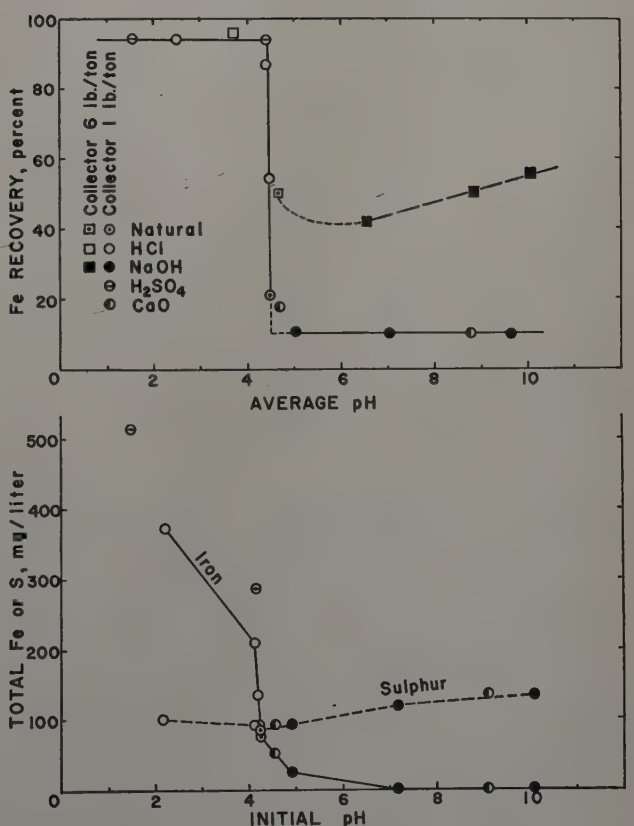


Fig. 14—Effect of pH on flotation recovery and total Fe and S in pulp solution, without activation. (a) flotation recovery, above: frother B-23, .04 lb per ton; collector amyl xanthate. (b) below: analysis of pulp solution before flotation.

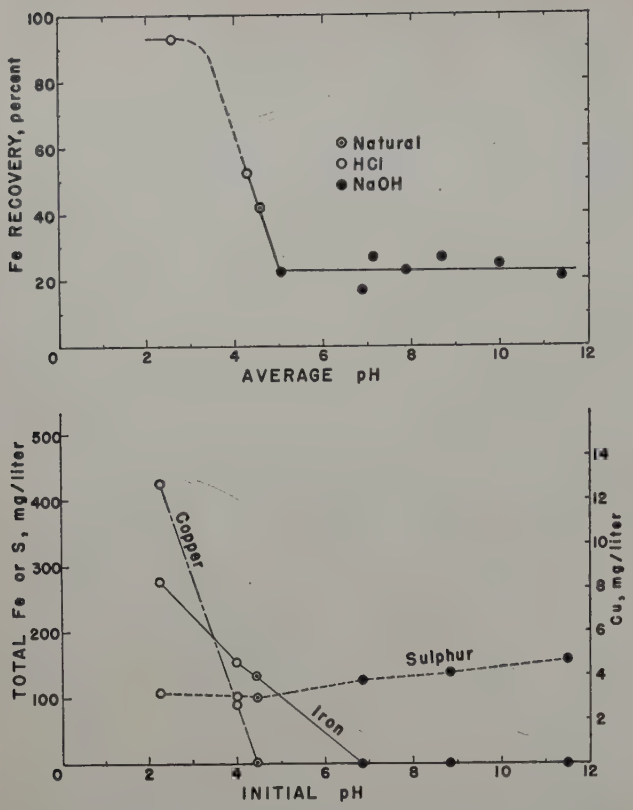


Fig. 15—Effect of pH on flotation recovery and total Fe, S, and Cu in pulp solution, with copper activation. (a) flotation recovery, above: frother B-23, .04 lb per ton; collector amyl xanthate, 1 lb per ton. (b) below: analysis before flotation.

Pyrrhotite has lattice constants of 3.43 Å along the a-axis and 5.79 Å along the c-axis.[12] The unit cell contains one iron atom in the (0001) plane, two in the $(10\bar{1}0)$ plane, and two in the $(01\bar{1}0)$ plane. Consequently there is one iron atom in a surface area of 10.2 $Å^2$ for the (0001) plane and 9.9 $Å^2$ for the $(10\bar{1}0)$ and $(01\bar{1}0)$ planes. Therefore, each iron atom occupies an average surface area of 10.0 $Å^2$. Since the pyrrhotite sample has a measured specific surface of 3000 cm^2 per g, there are 3×10^{18} iron atoms on the surface of every gram of this material, assuming that the sample is pure pyrrhotite. To obtain a complete monolayer replacement of iron atoms for a 300-g sample would require 254 mg of cupric chloride dihydrate. If the sites occupied by sulphur also adsorb copper, a maximum of 508 mg of cupric chloride would be required.

From Test No. A-8, omitting the initial loss of copper, 707 mg of cupric chloride equivalent of copper are abstracted by pyrrhotite. The replacement has extended beyond formation of a monolayer even if both sulphur and iron sites are considered to abstract copper, and the reaction would be very much beyond monolayer formation if initial loss of copper were taken into consideration. However, this does not take into account the effect of attrition that may take place in the Fagergren cell. An insubstantial increase in sulphur and iron upon further agitation and aeration after completion of replacement indicates that attrition is negligible. The change of the pyrrhotite specimens caused by copper activation in more concentrated solutions, shown in Fig. 8, is indicative of more than monolayer replacement.

The mineral surface is stabilized with the addition of 103 mg of cupric chloride, as shown in Fig. 5. That is, no iron and a negligible amount of sulphur pass into solution with extended aeration. Considering the theoretical minimum amount of 254 mg of cupric chloride required for formation of a monolayer, it is unnecessary to have complete monolayer replacement to stabilize the surface of pyrrhotite.

Several hypotheses have been presented to account for the adverse effect of oxidation of sulphide minerals on their flotation characteristics, but, in general, all of them can be included in the following two categories:

1—The detrimental effect of cations[13,14] or anions[15] formed by the oxidation of the minerals.

2—A change in the surface properties, which will inhibit the adsorption of collector on the mineral. Examples are the formation of a film on the mineral surface by either chemical reaction with, or adsorption of, some ions in the pulp.[16,17]

From the experimental work presented in Table IV, it can be seen that 1 hr of aeration of pyrrhotite with oxygen, air, and nitrogen before flotation resulted in recoveries of 29.3, 48.9, and 91.3 pct respectively, whereas the amounts of total iron and sulphur in the solution were essentially the same in all cases. Unless certain soluble sulphur compounds are detrimental to flotation, the effect of the soluble ions in the pulp can at present be regarded as a minor factor. Therefore, within the scope of the present investigation, differences in the flotation recoveries may be attributed to the formation of a surface film on the mineral. The aeration tests have shown that except for the initial stages, the sulphur increases at higher rate than the iron when oxygen or air is used, while both iron and sulphur increase at the same rate with nitrogen. This lower rate of liberation of iron than of sulphur in the case of air and oxygen aeration is suspected to be due to the precipitation of iron, presumably as ferrous or ferric hydroxide, some of which may coat the mineral surface. In Fig. 14b, the amount of iron in the pulp solution is seen to decrease rapidly with alkali addition, and the depression of pyrrhotite by this addition may be accounted for by the formation of a similar inhibiting coating on the surface.

Flotation tests have sharply defined the critical pH as about 4.5 for pyrrhotite when potassium amyl xanthate is used as collector. Wark,[18] using the contact angle method, reported the critical pH of pyrrhotite to be 6.0 in a solution containing 25 mg per liter of potassium ethyl xanthate. It is interesting to note that Yamada and Muraoka[19] report a critical pH of 4.6 to 5 for Yanahara pyrrhotite with a mixture of camphor blue oil and coal tar as collector.

It has been pointed out previously that the cleanliness of the mineral surface rather than the pH of the pulp may determine the floatability of pyrrhotite. This is further supported by the information given in Table IV. One hour of aeration with nitrogen gave a recovery of 91.3 pct at a pH of 5.1. On the other hand the oxygen-aerated pulp gave only 29.3 pct recovery when the pH was 4.2.

Reviewing the results of this investigation, it has been ascertained that the flotation circuit has to be acidified considerably to get a good recovery of pyrrhotite. However, this may not be practical if the ore contains calcareous minerals. In this case copper activation and stage addition of collector may provide a better solution of the problem.

Conclusions

The results of this investigation lead to the following conclusions.

1—Pyrrhotite in aqueous suspension liberates ferrous ion and soluble sulphur compounds, including sulphate ion.

2—Soluble ions pass into solution upon aeration with air, oxygen, and, after extended aeration, to a lesser extent with nitrogen.

3—A pulp consisting of pyrrhotite in water tends to reach an equilibrium pH.

4—Cupric ion is abstracted by pyrrhotite. The evidence indicates a stoichiometric exchange with iron atoms of the pyrrhotite.

5—A pyrrhotite surface can be stabilized by abstraction of less than a monolayer of copper.

6—Abstraction of copper can be more than is required for formation of a monolayer. The replacement of pyrrhotite by copper seems to be preferential along certain crystallographic directions.

7—Amyl xanthate is superior to ethyl xanthate as a collector for pyrrhotite.

8—Copper activation is beneficial to flotation of pyrrhotite, but excess cupric ion in the pulp is detrimental.

9—Stage addition of collector increases flotation recovery and requires less overall quantity of collector for equivalent recovery.

10—Acid addition greatly increases the floatability of pyrrhotite.

11—Pyrrhotite with potassium amyl xanthate as collector has a critical pH of 4.5. This corresponds roughly to the equilibrium pH obtained by aeration of a pulp containing pyrrhotite.

12—Copper activation is of no particular advantage in flotation beyond the critical pH.

13—Aeration with air and oxygen is detrimental to the flotation of pyrrhotite.

Acknowledgments

The authors are greatly indebted to Mr. V. E. Bye and his associates of the Mines Experiment Station, University of Minnesota, for conducting the many analytical determinations which were essential for this investigation and to Mr. C. E. Wood, Chief Metallurgist, Bureau of Mines, Region V, for the supply of the pyrrhotite sample.

References

[1] C. G. McLachlan: Twelve Months' Milling at Noranda. *Canadian Mining and Metallurgical Bulletin* (1930) **23**, pp. 237-258.

[2] I. N. Plaksin: On the Influence of Oxidation Processes on the Floatability of Sulphide Minerals. Comptes Rendus (Doklady) de l'Academie des Sciences de l'URSS (1945) **47**, No. 9.

[3] E. C. Herkenhoff: Separation of Pyrites, Arsenopyrites and Pyrrhotite by Flotation. Brit. Pat. 600,456 (1948).

[4] A. F. Taggart: *Handbook of Mineral Dressing*, pp. 12-110. John Wiley & Sons, Inc., New York, 1945.

[5] V. A. Malinovskii: Physicochemical Role of Air in the Flotation of Nonferrous Ores, I. *Tzvetnye Metal* (1938) No. 1.

[6] F. W. Bloecher, Jr.: A New Surface Measurement Tool for Mineral Engineers. *Trans.* AIME (1951) **190**, p. 225.

[7] E. B. Sandell: *Colorimetric Determination of Traces of Metals.* Interscience Publishers, Inc., New York, 1950.

[8] S. W. Parr: *The Analysis of Fuel, Gas, Water and Lubricants*, p. 260. McGraw-Hill Book Co., Inc., New York, 1932.

[9] C. S. Chang, S. R. B. Cooke, and R. O. Huch: Starches and Starch Products as Depressants in Amine Flotation of Iron Ore. *Trans.* AIME (1953) **196**, p. 1282.

[10] L. S. Foster: The Preparation of Xanthates and other Organic Thiocarbonates. Utah Eng. Exp. Sta. *T.P. 5* (1929).

[11] C. G. McLachlan: Synthetic Testing for Flotation. *Trans.* Can. Inst. Min. and Met. (1926) **29**, p. 262.

[12] E. H. Kraus, W. F. Hund, and L. S. Ramsdell: *Mineralogy*, p. 162. McGraw-Hill Book Co., Inc., New York, 1936.

[13] A. W. Hahn: Obviating the Harmful Effect of Soluble Salts in Flotation. *Engineering and Mining Journal* (March 12, 1952).

[14] A. B. Campbell, W. Howes, and W. H. Ode: Salts in Tri-State Mill Waters: Their Ill-Effect on the Flotation of Blende and Their Removal. U. S. Bur. Mines *R.I. 3149* (1932).

[15] J. F. Myers and F. M. Lewis: Use of a Conductivity Cell for Flotation Reagent Control. *Trans.* AIME (1946) **169**, p. 390.

[16] E. C. Plante and K. L. Sutherland: Effects of Oxidation of Sulphide Minerals on Their Flotation Properties. *Trans.* AIME (1948) **183**, p. 160.

[17] R. H. Richards and C. E. Locke: *Textbook of Ore Dressing*, p. 246. McGraw-Hill Book Co., Inc., New York, 1940.

[18] I. W. Wark: Principles of Flotation, p. 187. Australasian Institute of Mining and Metallurgy, Inc., Melbourne, 1938.

[19] G. Yamada and M. Muraoka: Effect of pH and Various Chemicals on the Flotation of Pyrrhotite. *Suiyokwai-shi* (1934) **8**, p. 435.

Botanical Methods of Prospecting for Uranium

by Helen L. Cannon

BOTANICAL methods of prospecting for metalliferous ores are based on the premise that deposits at depth may be reflected chemically in surface vegetation. For the past several years, on behalf of the Atomic Energy Commission, the U. S. Geological Survey has investigated on the Colorado Plateau the application of botanical methods to uranium prospecting. Results suggest that orebodies may be found under a considerable cover of barren sandstone through the absorption of uranium and associated trace elements by plants growing at the ground surface.

Plant physiologists have demonstrated that uranium is a necessary nutrient for plant growth and therefore is present in all plants.[1] The amount present is small but measurable. The absorption of uranium by plants growing over ore deposits forms

H. L. CANNON is a Geobotanist, U. S. Geological Survey, Washington, D. C.

Discussion on this paper, TP 3724L, may be sent (2 copies) to AIME before April 30, 1954. Manuscript, May 27, 1953. Los Angeles Meeting, February 1953.

the basis, therefore, for a method of prospecting by plant analysis.

Selenium and sulphur, two elements associated with uranium and vanadium in ores of many districts, are necessary in large quantities only to certain groups of plants. These plants, called indicator plants, are present only in soils or rocks containing large amounts of selenium and sulphur and can be mapped to indicate mineralized or geologically favorable ground. The distribution of indicator plants then offers a second tool for uranium prospecting.

Prospecting by Plant Analysis

Absorption of uranium by plants has been studied in connection with the development of botanical methods of prospecting.[2] Uranium is absorbed through the roots of plants, and much of the uranium is precipitated within the cells of the roots and held there; a lesser amount is transported to the branches and leaves. This amount in the branches and leaves is detectable, however, and is sufficient for reconnaissance prospecting. Uranium content varies with the species, time of year, and availability of the metal in the soil. It is rare for the uranium content in the ash of any species growing in nonmineralized sandstone to be greater than one part per million (ppm). A uranium content of several parts per million is common in the ash of plants rooted in ore. Any species of plant can be used in a systematic sampling program if that plant is widely distributed and the roots penetrate to the ore zone. The depth of root penetration differs for any given area with the species of plant and the depth to water. Deep-rooted shrubs and trees of semi-arid regions commonly have root systems 30 to 50 ft in length, and roots have been observed in mines at depths as great as 100 ft.

A basic problem associated with the collection of large numbers of samples is the establishment of a sample type that is uniform, representative, easily procured, and easily handled. Deep-rooted junipers and piñons fulfill these requirements most satisfactorily on the Colorado Plateau, although shadscale and other low shrubs may be used in areas where ground water is not available to vegetation. A composite sample of branch tips or leaves is taken from all sides of the tree and at the same distance aboveground on the tree. A two-man party can collect 50 samples per day spaced at intervals of a few hundred feet. Rapid laboratory methods of analysis, however, have not yet been perfected to handle large numbers of samples on a comparable daily basis. At present the samples are put through an ashing process, a chemical extraction, and finally a fluorimetric analysis using a fluoride-carbonate flux.[3] Samples are collected during the field season for chemical analysis in Survey laboratories during the winter months.

Artificial contamination introduces a considerable source of error in sampling areas of active mining or milling. Dust from an operating surface pit may raise the uranium content of nearby trees from 2 to 100 ppm in the ash. Contamination from the mill at Uravan, Colo., has substantially increased the content of uranium in juniper ash for a distance of several miles. A sampling program on the dune sands in the Carrizo Mountains of Arizona resulted in outlining only the roads used by ore trucks.

The discussion so far has been concerned only with the absorption of uranium from carnotite (potassium uranyl vanadate) deposits occurring in the Colorado Plateau. Uranium is more readily absorbed by plants growing on a carbonate deposit like schroeckingerite (a complex uranyl carbonate-sulphate) than on carnotite. Seven thousand ppm, or seven-tenths of a percent in the ash was found in roots of a greasewood plant growing in a deposit of schroeckingerite in the Red Desert in Wyoming. The uranium content of trees growing on the limestone deposits of Grants, N. M., is large where the deposits have been exposed to weathering. Apparently absorption of uranium by plants is increased in the presence of soluble carbonates.

A plant may be used as an indicator in botanical prospecting if its distribution is controlled by any factor related to the chemistry of the ore deposit. In carnotite districts, the absence of specific groups of plants is as indicative of mineralized ground as the presence of certain others. In arid soils of high salt content, for instance, the common saltbush, sagebrush, and hop sage are scarce or even absent. They are generally replaced by shadscale, turpentine weed, and rabbitbrush. This replacement effects an overall change in the appearance of the flora and is a common characteristic of carnotite districts.

Prospecting by Mapping Indicator Plants

In a specific search for orebodies within a given district, selenium- or sulphur-indicator plants may be very useful. Selenium-indicator plants bear a recognizable relationship to carnotite deposits of the semi-arid Southwest. The various species on and around deposits have distinct distribution patterns, which depend on the amount of selenium in the ore and the amount required by the plant. For this reason some species are more important than others in prospecting for mineralized ground in a given locality. Plants that require large quantities of sulphur were at first ignored in this study. However, recent drilling in the Thompsons district, Utah, has shown them to be useful in indicating ores that contain an appreciable quantity of sulphates. The plants belong generally to three families, lily, mustard, and buckwheat. The same or closely related plants may also be used as indicators of sulphide ores in base-metal districts. Six sulphur-indicator plants are shown in Figs. 1-6.

The onion and Sego-lily, both of the lily family, are very shallow-rooted and therefore are useful only where sulphates have migrated upward into the surface soils. The degree of upward migration in arid soils was demonstrated in the Thompsons district, Utah, where several orebodies at an average depth of 20 ft were discovered under indicative patches of these plants. Desert trumpet, an *Eriogonum* with an inflated stem, is tolerant of high-sulphate clay soils. It is common around gypsiferous uranium ores and has been used in locating wagon-drill holes in some areas. Many other species of *Eriogonum* act as indicator plants near uranium and sulphide ores. Peppergrass and other members of the mustard family are common on near-surface ores. Tumbleweed mustards are abundant around ore deposits on some of the higher mesas. *Stanleya*, or prince's plume, is a mustard that requires both selenium and sulphur for growth.

The selenium-indicator plants require a definite amount of selenium to maintain growth. However, the amount required is known to differ with the different species, and each plant develops a distinctive distribution pattern around a given deposit. Six selenium-indicator plants are shown in Figs.

Sulphur-Indicator Plants Common in Uranium Districts, Figs. 1-6

Sisymbrium altissimum (tumble mustard)

Toxicoscordion gramineum (death camas)

Eriogonum inflatum (desert trumpet)

Lepidium montanum (peppergrass)

Stanleya pinnata (prince's plume)

Allium acuminatum (wild onion)

Selenium-Indicator Plants Common in Uranium Districts, Figs. 7-12

Astragalus confertiflorus (milk vetch)

Astragalus preussi var. arctus (milk vetch)

Astragalus thompsonae (milk vetch)

Astragalus pattersonii (milk vetch)

Oryzopsis hymenoides (ricegrass)

Aster venusta (woody aster)

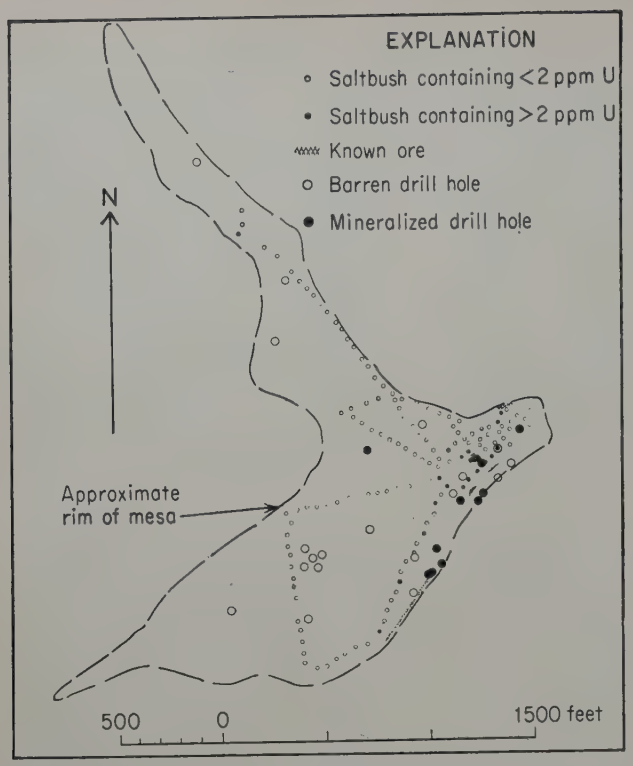


Fig. 13—Sketch map of Yellow Cat mesa, Grand County, Utah, showing distribution of uranium content in saltbush in relation to mineralized ground.

7-12. Woody asters and ricegrass have low selenium requirements and are common on alluvium and dune sands in carnotite districts. Certain species of *Astragalus,* or milk vetch, are the most effective indicators in carnotite districts. The genus is a large one containing hundreds of species, but only certain tribes of them are selenium-absorbers and only a comparatively few species are useful in prospecting. Several of the species are small rosette forms difficult to distinguish or to map on a large scale. *Astragalus thompsonae* with furry pods is one of these. *Astragalus confertiflorus* has blue foliage and grows almost exclusively on blue mudstone, where the plant extracts large quantities of ferrous iron. The selenium requirements for this plant are low, but the plant is useful in prospecting favorable portions of an ore-bearing outcrop. The most useful indicators of uranium deposits are closely related, belong to the same tribe, and are somewhat difficult to tell apart. Two species occurring in the Thompsons district are *Astragalus preussi* and *Astragalus pattersonii.* The flowers of *A. preussi* are pink; and the flowers of *A. pattersonii* are creamy white. *A. pattersonii* grew around 13 holes out of a total of 26 ore holes drilled in the Yellow Cat area in 1952.

Evaluation of Botanical Methods

Prospecting by plant analysis is limited to detecting anomalous material within about 50 ft of ground surface. In a few places, where jointing is well developed, tree roots penetrate sandstone beds 60 to 70 ft thick to reach ground water. Where the ore-bearing bed is also an aquifer, the absorption of uranium by trees is uniform and the resulting uranium values can be contoured.

The accuracy of predicting mineralized ground is of a high order. More than 2 ppm uranium in trees is indicative of geologically favorable ground and usually indicative of mineralized ground. An example is shown in Fig. 13. Several hundred samples of shadscale were collected on a mesa in the Thompsons district. The only botanically favorable ground is in the southeast corner between two known orebodies. Drilling proved the mesa to be barren except in the indicated section, where two orebodies were discovered. During the 1952 field season, more than 3000 samples of juniper and piñon were collected on a bench of the Todilto limestone member of the Wanakah formation near Grants, N. M. The results have been contoured section by section and turned over to the Atomic Energy Commission in advance of drilling. Although the area has not yet been drilled by the Atomic Energy Commission, many of the anomalies which have been explored by private enterprise have been found to be mineralized.

Prospecting by indicator plants has been tested in the Thompsons district, where plants were mapped over an area of 6 sq miles and where 1000 holes were subsequently drilled. A summary of results is shown in Table I. The restriction of indicator plants to geologically favorable ground was found to be an important guide. Of the 194 holes drilled in areas supporting the growth of indicator plants, 90 were in mineralized ground. Indicator plants are present in the vicinity of 12 pct of the unmineralized holes, 54 pct of the mineralized holes (including ore holes), and 81 pct of the ore holes. Four orebodies of the ten discovered in the Yellow Cat area would not have been found by means of the grid pattern of exploration drilling had it not been for the additional information furnished by indicator plants.

Table I. Correlation of Indicator Plants with Mineralized Ground in the Thompsons District, Utah

Item	Unmineralized Holes	Mineralized Holes		Ore Holes		Totals	
		No.	Depth to Ore Horizon, Ft	No.	Depth to Ore, Ft	No.	Depth to Ore Horizon, Ft
Plants present	104	69	44	21	25	194	40
Plants absent	731	70	59	5	37	806	57
Total holes drilled	835	139	53	26	27	1000	49
Ratio of botanically favorable holes to total no. of holes		12 pct		54 pct		81 pct	

Results of drilling demonstrate that holes must be placed with close reference to plant patches and with careful regard for dip, ground-water movement, joint patterns, and other geologic features. In many places several holes were drilled in the vicinity of a plant patch to discover the orebody out of which the selenium had moved. Although the statistical score is imperfect, botanical prospecting is probably a worthwhile tool in the search for uranium and is useful wherever deposits are less than 70 ft from surface and where selenium and uranium are readily available to plant roots.

References

[1] Julius Stoklasa and Joseph Penkava: Biology of Uranium. *Zeitschrift Biochemie* (1928) **194**, pp. 15-77.

[2] H. L. Cannon: The Effect of Uranium-Vanadium Deposits on the Vegetation of the Colorado Plateau. *American Journal of Science* (1952) **250**, pp. 735-770.

[3] F. S. Grimaldi, May Irving, and M. H. Fletcher: U. S. Geological Survey Fluorimetric Methods of Uranium Analysis. U. S. Geol. Survey *Circ. 199*, 13 pp.

Flotation Theory: Molecular Interactions Between Frothers and Collectors at Solid-Liquid-Air Interfaces

by J. Leja and J. H. Schulman

FROTH flotation is usually effected by the addition of a collector agent and a frothing agent to an aqueous suspension of suitably comminuted mineral ores. The action of collectors is to adsorb onto the surfaces of minerals to be separated, sensitizing them to bubble adherence. The action of frothers has, in the past, been accepted as that of froth formation only, brought about by a lowering of the air/water interfacial tension. Substances capable of producing froth are classed[3a, b] according to their relative capacities for production of froth-volume and froth stability in the simple frother-water system.

The purpose of this paper is to show that the surface active agents acting as frothers become effective only when there is a suitable degree of molecular interaction taking place between collector molecules and frother molecules at the air/water and solid/water interfaces. Further, the discussion will demonstrate that the actual mechanism of adherence of an air bubble to a suitably collector-coated particle is due to the molecular interaction collector-frother. This leads to the formation of a continuous interfacial film of associated molecules, anchored to the mineral by polar groups of the collector, and enveloping the whole bubble. The tenacity of adhesion mineral-to-bubble results from the strength and the visco-elasticity of this mixed film.

Some 20 years ago Christman[2] postulated mutual dependence of collector and frother in effecting flotation. This view was, however, strongly opposed by Wark,[3] who pointed out that an addition of frother had no effect on the value of contact angle once this was established in the solution of collector. More recent work by Taggart and Hassialis[4] indicated that the presence of frother, namely, cresol, leads to the immediate establishment of a contact angle on sphalerite, partially coated with xanthate, whereas an air bubble fails to make contact in potassium ethyl xanthate solution alone, even after 60 min induction time. Wrobel[5] draws attention to the selectivity of frothers in flotation.

Many instances of antagonistic effects of certain mixtures of frothers (or collectors and frothers) on flotation froth have been known to flotation operators and have been reported in literature. Taggart[6] and Cooke[7] give several examples of incompatibility of certain ratios of frothers and collectors, e.g., oleate and long-chain sulphates, pine oil and soaps.

Monolayer Penetration. Properties of insoluble films produced by molecules of surface active agents orientated at the air/liquid interface are conveniently studied by the Langmuir trough technique, described fully by Adam.[8] Using the trough technique Schulman and Hughes[9] and Schulman et al.[10a, b, c, d, e] established the existence of molecular interactions occurring between certain types of surface active agents. Their experiments revealed the phenomenon of penetration of an insoluble monolayer (e.g., a film of a long-chain alcohol) by a soluble agent (e.g., sodium alkyl sulphate) injected into the substrate (water or salt solution). The degree of molecular interaction taking place on penetration is determined by changes in the surface pressure of the resulting film, changes of its surface potential and its mechanical properties (viscosity and rigidity).

When the interaction takes place between both polar groups and both hydrophobic groups of the two participating amphipathic molecules a molecular complex is formed.

Complexes formed on penetration of the monolayer at interfaces are not necessarily true chemical compounds: they are labile in solution, the activity and reactivity of individual components are greatly different from those of the molecularly associated complex, and on crystallization they usually separate out into components. However, when formed in the orientated state at interfaces they are found to be very stable, although some mixed films spread as monolayers of stoichiometric complexes can show further penetration by subsequent additions of the soluble component injected into the substrate.[10e]

The degree of association between two or more types of surface active agents is very sensitive even to small changes in electric (dipole) moment of the polar groups of the amphipathic molecules as influenced by magnitude and position of neighboring ions or dipoles, their size, concentration, and stereochemistry. In addition, the molecular association is greatly influenced by concentration and type of inorganic salts in the substrate, by its pH, and by temperature. The nonpolar groups of interacting molecules greatly affect the stability of molecular complexes. Progressive shortening of the aliphatic chain of one of the reacting molecules weakens (at an increasing rate) its tendency to form stable complexes. Similarly, introduction of a double bond of cis-form into one of the reacting chains, which changes the straight hydrocarbon chain into a kinked one, or introduction of a branched chain, reduces the stability of the associated complex.

Monolayer Adsorption. Using the trough technique and injecting metal ions into the substrate (water or salt solution) underlying insoluble films of fatty acids, alkyl amines, and sulphates, Wolstenholme and Schulman[11a, b, c] and Thomas and Schulman[12] have established conditions, namely, pH, concentration, and steric factors, under which molecular interactions take place between the *polar groups* of the surface active agents and the *metal ions.* These interactions are marked by great changes in the solubility and mechanical properties of the monolayer of the agent; no surface pressure increases are observed as in monolayer penetration experiments. The results of these adsorption studies, correlated with flotation experiments, indicated that in the case of fatty acids and alkyl sulphates their adsorption onto minerals of base-metals takes place by a similar

J. LEJA, Member AIME, is Research Engineer, Ernest Oppenheimer Laboratory, Department of Colloid Science, The University, Cambridge, England. J. H. SCHULMAN is Reader in Surface Chemistry, The University, Cambridge, England.

Discussion on this paper, TP 3693B, may be sent (2 copies) to AIME before April 30, 1954. Manuscript, Aug. 18, 1953. New York Meeting, February 1954.

mechanism. Flotation of these minerals occurs whenever the pH and the size of metal ions available at the mineral surface enables the formation of an insoluble film of the *basic-metal soap.* The films of the agents are solidified by the adsorption process owing to hydrogen bonding between the adlineated hydroxyl groups of basic-metal ions; the van der Waals forces between *widely spaced* hydrophobic groups are negligible.

Experimental procedure was based on: (a) interactions between an insoluble alcohol or sulphate monolayer, short-chain homologues of which are used as frothers, spread on the Langmuir trough, and a xanthate (typical collector) injected into the substrate; (b) determinations of the amount of frother abstracted from solution by mineral particles coated with collector; (c) measurements of contact angles formed by air bubbles and insoluble oils on solid surfaces in collector solutions; and (d) frothing and flotation tests using equivalent additions of collectors and frothers.

All reagents used in experiments were of high purity; they were either made and purified in the laboratory (xanthates, oleic acid, alkyl sulphates and sulphonates, alkyl amine hydrochlorides, and substituted amines) or were high-purity commercial products (alcohols). The latter were checked for impurities, capable of adsorption on solids, by contact angle testing on a highly reactive surface of electrolytically polished copper. Only those alcohols were used which did not develop any contact angle, i.e., which contained no impurities of collector-type.

(a) Trough interactions. It can be readily shown that by monolayer penetration of long-chain alcohol films by long-chain xanthates, strong molecular associations exist between these two types of compounds. If a monolayer of hexadecyl alcohol (cetyl alcohol, C_{16} H_{33} OH) is spread on N/100 KCl solution at pH 11 and is held at a constant area and at a surface pressure of 10 dynes per cm, and ½ mg of potassium lauryl xanthate (C_{12} H_{25} xanthate) is then injected into the underlying solution of 400 cc, *an immediate rise of surface pressure of the alcohol film from 10 dynes per cm to 52 dynes per cm takes place,* although the potassium lauryl xanthate solution itself shows initially only 5 dynes per cm surface pressure. This surface pressure of the free lauryl xanthate monolayer, initially equal to 5 dynes per cm, decays to zero pressure rapidly owing to the oxidation of xanthate into dixanthogen molecules, which form only unstable films on their own. The mixed monolayer alcohol-xanthate *remains stable* to very high surface pressures, e.g., 52 dynes per cm. This indicates that the *penetrated alcohol-xanthate monolayer stabilizes the xanthate against oxidation.*

Weak penetrations can still be observed with amyl xanthate (C_5) into cetyl (C_{16}) alcohol monolayers. This type of association would show up more readily with surface potential measurements. The cetyl alcohol film is liquid but on penetration by lauryl xanthate is immediately solidified.

Penetration of a long-chain sulphate monolayer by lauryl xanthate is much weaker than alcohol-xanthate penetration. An increase in surface pressure up to 25 dynes per cm, with 2 mg per 400 cc, takes place with a sodium behenyl sulphate monolayer (C_{22} H_{45} SO_4 Na). Like the cetyl alcohol-xanthate monolayer, the behenyl sulphate-xanthate monolayer is solidified, but much more slowly.

These experiments show that the long-chain xanthates are much more reactive to long-chain alcohols than to the equivalent long-chain sulphates.

(b) Abstraction of Frothers by Collector-Coated Solid Particles (Table I). Procedure: A quantity of air-atomized Cu powder or pure mineral particles was shaken in a cylinder with a solution of collector. Then a solution of frother was added either directly to the cylinder or after prior removal of collector solution by decantation. Comparative determinations of suitable dilutions of frother with distilled water alone, the collector solution, and the residual solution in the cylinder indicated the amounts of frother abstracted by collector-coated particles. Blank determinations for abstraction of frother by solid particles not coated by collector were carried out and showed negligible amounts of abstraction.

Colorimetric methods of analysis for aliphatic alcohols and cresols were adapted from Snell.[13] α Terpineol was determined by the intensity of the colored compound given with salicylic aldehyde, in 66 pct H_2 SO_4 solution; cresols, by the colored compounds given with diazotized sulphanilic acid in NaOH solution. Intensities of color were compared using a Spekker absorptionmeter. With the very low concentrations of frothers used in flotation, extreme care is required in analytical procedure to get reliable and reproducible results. In addition, steps must be taken to avoid interference due either to collector itself or to the products of collector decomposition. Xanthates decompose initially to dixanthogens and finally to alcohols and carbon disulphide. Determinations of alkyl alcohol abstraction cannot, therefore, be reliably carried out in systems containing xanthates. Cresols can be determined only when excess xanthate is removed prior to conditioning with frother.

Determinations of long-chain sulphates and sulphonates were carried out by the partition titration (in chloroform-water) with cetyl trimethyl ammonium bromide (CTAB), using methylene blue as an indicator. The partition titration of long-chain anionic compounds with cationic salts was developed by Hartley and Runnicles[14] and Epton.[15] Owing to hydrolysis of alkyl sulphates only sulphonates could be used in abstraction tests of xanthate-coated solids.

Determinations of frother abstraction were reproducible to 1/10 to 1/5 of *the amount abstracted.* Accuracy of the analytical methods was of the order of 0.001 mg per ml for cresols, 0.01 mg per ml for α Terpineol, 0.005 mg per ml for sodium dodecyl sulphonate.

Abstraction results: Cuming and Schulman[16] have found that adsorption of sodium dodecyl sulphate onto copper powder is possible in the narrow range of pH 5.5 to 9.0 where the insoluble basic copper sulphate soap (C_{12} H_{25} SO_4 (OH) Cu) is formed. In the more alkaline solution the surface of Cu powder is incapable of adsorbing sulphate, but xanthates are still strongly adsorbed.

Sulphonates and sulphates behave similarly in flotation of Cu powder. The alkyl sulphonate gives excellent flotation of Cu at pH 5.5 to 9.0, acting both as a collector and frother, but once the pH is increased above 9.0 the sulphonate starts desorbing, leaving Cu powder completely *hydrophilic.*

Sulphonate concentrated in solution gives excessive but barren froth. If xanthate is now added to the system containing Cu powder and sulphonate at pH $>$ 9, say pH 12.5, it adsorbs immediately on Cu powder and excellent flotation is again effected. At the same time the excessive, barren froth of sulphonate evident in absence of xanthate is replaced by a well-mineralized, relatively stable froth.

Tests 1 and 2 of Table I show that Cu powder

coated by potassium amyl xanthate (KAX) abstracts molecules of sodium dodecyl sulphonate (C_{12} H_{25} SO_3 Na) at pH 12.5. The blank for abstraction of sulphonate by Cu powder, not sensitized by xanthate-coating, was nil under the same conditions. It can be seen that in test 1 the initial concentration of sulphonate has been reduced by 18 pct, equal to 0.490 mg sulphonate abstracted by 50 g of −300 mesh Cu powder. Test 2 showed 27 pct reduction of initial concentration, equal to 0.387 mg abstracted by 50 g of −100 +200 mesh Cu powder.

Notwithstanding the longer time of conditioning with both collector and frother, the abstraction of sulphonate is lower in test 2 than in test 1, owing to the smaller area of particles: ca 80 cm^2 per g for −100 +200 mesh, and ca 400 cm^2 per g for −300 mesh Cu powder.[17]

Tests 3 and 4 refer to the abstraction of p-cresol by Cu powder sensitized with dodecyl sulphonate at pH 7.5. Although sulphonate acts at this pH as both collector and frother, once the sulphonate remaining in solution and acting as frother is decanted, the floatability of sulphonate-coated particles in distilled water is negligible, unless a frother such as cresol is added. The froth produced by cresol is less voluminous and more brittle than the excessive, tough froth due to sulphonate itself, but the flotation recovery of Cu is not affected. Comparison of tests 3 and 4 shows the effect of time on the degree of p-cresol abstraction, under similar conditions. It is seen that the abstraction of p-cresol continued to increase with the time of conditioning. This effect of time is an indication of a relatively slow rate of molecular association of sulphonate and p-cresol.

Tests 5 and 6 show that abstraction of cresols does take place even when a short-chain xanthate (potassium ethyl xanthate) is employed as collector. Further, they indicate that the amounts abstracted vary by a factor of 10 at least, depending on initial concentrations of collector and frother and the surface area of particles.

Test 8 shows that with reagents which are known to give strong complexes in stoichiometric ratio the

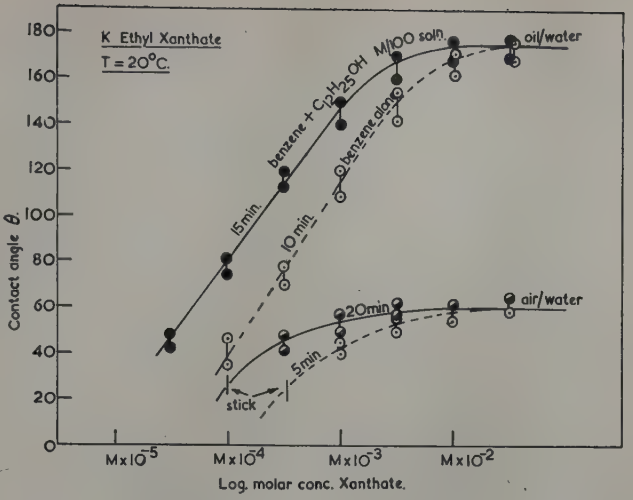


Fig. 1—Relationship: contact angle-concentration of ethyl xanthate on electrically polished copper surface; pH 8.6, temp. 20°C; effects of time of conditioning and of presence of complexing alcohol in the oil phase.

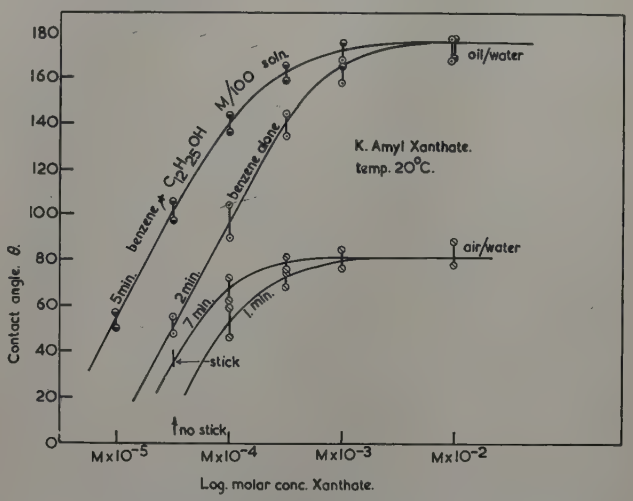


Fig. 2—Contact angle-concentration of amyl xanthate (conditions as in Fig. 1).

Table I. Results of Analytical Determinations of Abstractions of Frothers. Temperature 20°C ± 2°

Test No.	Material	Collector			Frother			pH	Abstraction of Frother	
		Compound	Conc.	Time of Cond.	Compound	Conc.	Time of Cond.		Amount Abstracted	Initial Frother Conc., Pct
1	50g of −300 mesh Cu (a)	KAX (c)	M/1300 2:1 S:W	15 min	$C_{12}H_{25}SO_3$ Na	M/7500 2:1 S:W final 1:1 S:W	10 min (c)	12.5	0.490 mg per 50g Cu	18
2	50g of −100 + 200 mesh Cu (a)	KAX (c)	M/10000 2:1 S:W	2 hr	$C_{12}H_{25}SO_3$ Na	M/10000 2:1 S:W final 1:1 S:W	1 hr (c)	12.5	0.387 mg per 50g	27
3	50g −300 mesh Cu (a)	$C_{12}H_{25}SO_3$ Na (c)	M/2500 1:1 S:W 26 pct ads	10 min (d)	p-cresol	M/2500 1:1 S:W	10 min (c)	7.5	0.450 mg per 50g	21
4	50g −300 mesh Cu (a)	$C_{12}H_{25}SO_3$ Na (c)	M/2500 1:1 S:W 28 pct ads	10 min (d)	p-cresol	M/2500 1:1 S:W	5 hr (c)	7.5	0.685 mg per 50g	31
5	20g PbS, −60 +200 mesh	KEX	M/20000 1:1 S:W	30 min	o-cresol	M/20000 final 1:2.5 S:W	10 min *cell test*	7.5	0.056 mg per 20g	22
6	50g ZnS −60 +100 mesh (b)	KEX (c)	M/12500 1:1 S:W	10 min (d)	o-cresol	M/1250 1:1 S:W	2 hr (c)	7.5	1.54 mg per 50g	35
7	30g $BaSO_4$ −120+ 300 mesh	$C_{12}H_{25}SO_3$ Na (c)	M/10000 1:1 S:W 36 pct ads	15 min (d)	αTerpineol	M/5000 1:2 S:W	4 hr (c)	8.0	2.2 mg per 30g	35
8	50g Cu −300 (a)	$C_{12}H_{25}SO_3$ Na (c)	M/1000 1:1 S:W 46 pct ads	20 min (d)	CTAB	M/10000 1:1 S:W	15 min (c)	7.0	1.092 mg per	48

(a) air atomized Cu powder
(b) sphalerite containing 3.1 pct Cu_2S and 15 pct pyrite
(c) cylinder test
(d) solution of collector decanted

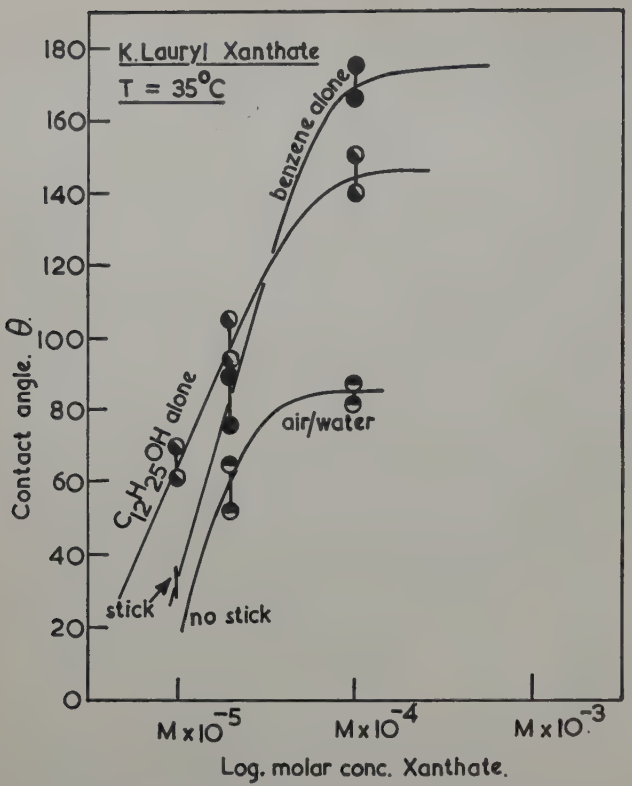


Fig. 3—Contact angle-concentration of lauryl xanthate, on copper surface, pH 8.6; effect of polarity of oil.

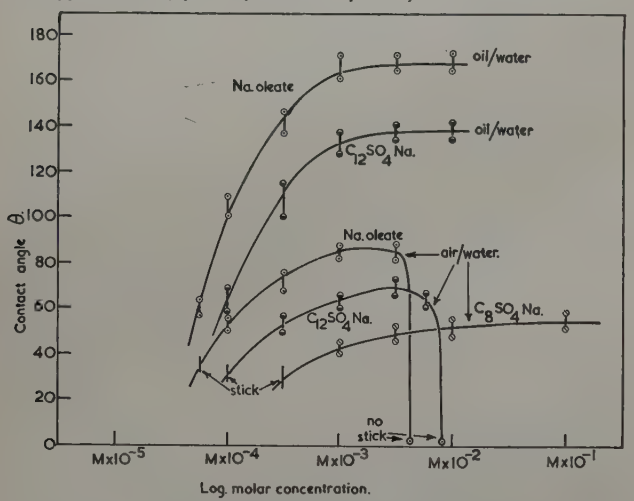


Fig. 4—Relationship: contact angle-concentration of carboxylic soaps and alkyl sulphate, on cleaved $BaSO_4$ surface; pH 12.0, temp. 20°C, conditioning time 5 to 10 min.

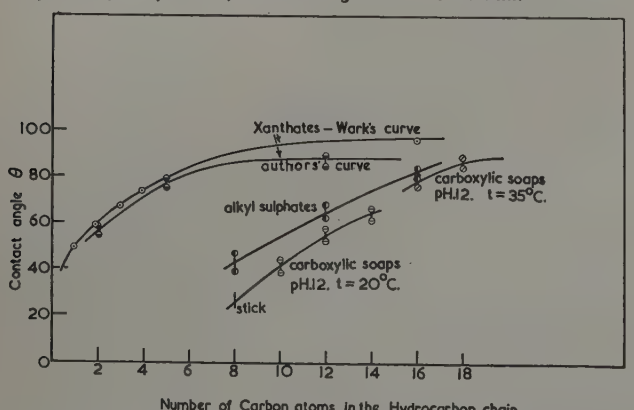

Xanthates – Wark's curve
authors's curve
carboxylic soaps pH.12. t = 35°C.
alkyl sulphates
carboxylic soaps pH.12. t = 20°C.
stick
Contact angle θ
Number of Carbon atoms in the Hydrocarbon chain.

Fig. 5—Relationship: contact angle-chain length of collector, for M/1000 solutions; pH 12.0 for carboxylic soaps, pH 8 to 12 xanthates and alkyl sulphates, temp. 20°C (and 35°C), conditioning time 5 to 10 min.

abstraction proceeds to give nearly a 1:1 relation between collector coverage and frother abstraction. As equimolar concentrations of collector and frother were used, the comparison may be made directly on percentage of initial concentration removed by particles: 46 pct of adsorbed sulphonate abstracted 48 pct of CTAB. Test 7 indicates that the ratio for α Terpineol : sulphonate is nearly 2:1; 35 pct of M/5000 α Terpineol solution was abstracted by 36 pct of M/10000 adsorbed sulphonate.

(c) Contact Angle Measurements. Procedure: A horizontal telescope equipped with cross wires and protractor was used; contact angles were always measured across the water phase. In addition to air bubbles, drops of various oils such as benzene, alcohols, and nujol were also employed. The usual experimental precautions of cleaning the glassware in chromic-sulphuric acid mixture and carrying out blank determinations in distilled water were observed. The contact angle could be measured with the accuracy of ± 1°, but owing to hysteresis, the angles varied by 5° to 10°.

Solid surfaces used for contact angle measurements consisted of freshly electrolytically polished specimens of copper bar (anodic polishing in 55 pct H_3PO_4 solution) and freshly cleaved faces of $BaSO_4$ crystals. Conditions for measurements were standardized with regard to time of conditioning, time of bubble-contact, and *aging* of the reactive surfaces.

Figs. 1 to 6 and Table II. Fig. 1 shows the effects of the concentration, the time of conditioning, and the presence of an alcohol in the oil phase on the development of contact angles between the air/water and oil/water interfaces and the solid surface of electrolytically polished copper metal specimen for potassium ethyl xanthate (KEX). It is seen that for 5 min conditioning time the air/water/solid contact angle can be at first detected as a pronounced *stick* of the air bubble (at concentration of 0.5×10^{-3} KEX), and then progressively increases to a maximum of 60°, with the rise in concentration. For 20 min conditioning time, the development of contact angle starts at a much lower concentration, viz, M x 10^{-4}, and the maximum of 60° is reached sooner. The oil/water/solid contact angles (using pure benzene as the oil phase) can be detected at much lower concentrations of xanthate than those required for air/water contact angle development. At the same concentrations of KEX, the oil/water/solid contact angles are much higher in value than the air/water/solid ones and reach the maximum of nearly 180°, i.e., the benzene drop spreads nearly completely on the xanthate covering the solid surface. If instead of a benzene drop, a drop of M/100 solution of lauryl alcohol (C_{12} H_{25} OH) in benzene is used, the contact angles developed at equal concentrations of xanthate are still higher than those for benzene alone, and the rate of adsorption is much greater.

These curves show clearly that the development of the contact angle depends on the combined effects of the concentration of xanthate and the time of conditioning. These in turn *govern the density of adsorption* of xanthate molecules onto the solid, i.e., the collector coverage. Further, it is shown that the effect of a complexing agent, alcohol, is evident only when collector *diffusely covers* solid surface.

Fig. 2 shows that a similar relationship exists for potassium amyl xanthate; the range of concentrations for which the contact starts to develop is lower than in the case of potassium ethyl xanthate, the maximum of the air/water/solid contact angle is

higher, viz, 80° to 82°, but the maximum of the oil/water/solid is the same as for potassium ethyl xanthate.

Fig. 3 shows the same characteristic developments of contact angles for potassium lauryl xanthate, at still lower concentrations than for potassium ethyl xanthate, and with a still higher maximum of the air/water/solid contact angle. In addition, the relationship between the concentration of lauryl xanthate and the contact angles developed by a drop of lauryl alcohol alone is also shown. It is seen that when the surface of copper is sparsely covered by lauryl xanthate molecules (i.e., in concentrations lower than 0.5 M x 10^{-4}) the drop of alcohol develops higher contact angles than the drop of benzene; at higher concentrations of xanthate, the maximum contact angle is, however, only 145° for alcohol alone, whereas with benzene it is 180°. This behavior indicates that with the partly-hydrophobic surface, diffusely covered by xanthate molecules, the complexing molecules of alcohol can penetrate the diffuse monolayer and thus increase the hydrophobic character of the surface. However, once the full coverage is attained by the collector molecules themselves the penetration is prevented, and the polarity of alcohol molecules in relation to benzene is demonstrated by the differences in contact angles.

Fig. 4 shows the relationship between contact angles on Ba SO_4 crystal faces and concentration of collectors: sodium oleate (cis $CH_3(CH_2)_7CH = CH(CH_2)_7COONa$), sodium dodecyl sulphate ($C_{12} H_{25} SO_4 Na$), and sodium octyl sulphate ($C_8 H_{17} SO_4 Na$).* These types of surface active agents (i.e., carboxylic acids and their soaps, alkyl sulphates, also alkyl sulphonates, alkyl amines and their salts) are capable of adsorption as orientated molecular films, at both the solid surfaces and the air/liquid interface. They are referred to, for flotation purposes, as collector-frother agents. Xanthates (as well as dithiophosphates and dithiocarbamates) adsorb at the air/liquid interface but are immediately oxidized to insoluble dixanthogens, which consequently do not froth. The surface active agents of the collector-frother type are not appreciably affected by oxidation, but they *associate into micelles of hydrophilic character* at high concentrations. For particular polar groups the longer the chain of the surface active agents the lower is the critical concentration (C.M.C.) for micelle formation. It is important to bear in mind that this C.M.C. is influenced by solutions of inorganic salts.

* Further data on contact angles for other soaps and sulphates may be found in the authors' paper: Control of contact angles at the oil/water/solid interfaces in relation to emulsions stabilized by solid particles. *Trans.* Faraday Soc., 1954.

Table II. Effect of Di-carboxylic Soaps on Ba SO_4

Soap M/1000 soln	pH	Contact Angle: Air/Water	Contact Angle: Benzene/Water	Flotation Tests in Pneumatic Cell
Na-azelaate Na+' $OOCC_7 H_{17}COO'$ Na+	7 to 12	No sticking	No sticking	No flotation
Na thapsate	7.0	Slight sticking	40° to 45°	Very good flotation
Na+' $OOC C_{14}$	9.0	Good sticking	60° to 65°	
$H_{28} COO'Na+$	12.0	Very good sticking but impossible to detach a free air bubble	75° to 80°	

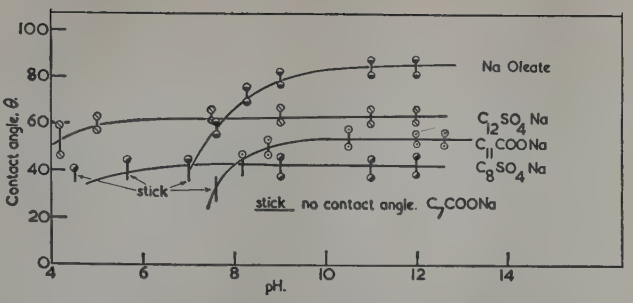


Fig. 6—Relationship: contact angle-pH; M/1000 solutions of carboxylic soaps and sulphates; temp. 20°C, conditioning time 5 to 10 min.

The immediate effect in flotation of the formation of micelles by carboxylic soaps, alkyl sulphates, and also amines is the nonadherence of air bubbles to the solid surface coated by these agents in solutions of high concentrations. The proof that the surface is then still coated by the surface active agent is furnished by the development of full contact angles at the oil/water/solid interfaces. Oil is capable of penetrating the physically bound hydrophilic double layer on the solid and gives as high a contact angle as the fully hydrophobic single layer. These effects are shown in Fig. 4 for Na oleate and Na dodecyl sulphate. The short-chain sodium octyl sulphate ($C_8 H_{17} SO_4 Na$) shows a comparatively weak tendency for association, and its critical micelle concentration is higher than M x 10^{-1}, but sodium dodecyl sulphate and sodium oleate form micelles at concentrations less than M x 10^{-2}.

Figs. 5 and 6 show the effects of the length of chain and pH on the contact angles at the air/water/solid interfaces. From Fig. 5 it is apparent that for a given concentration of the reagent it is not only the length of chain itself but also the nature of the polar group (sulphate, xanthate, or carboxyl) that determines the magnitude of the contact angles developed with solids capable of adsorbing the reagent.

As is well known, the same agent adsorbed on different solid surfaces gives the same maximum values of contact angles.

Table II shows the results of adsorption on Ba SO_4 crystal of di-carboxylic soaps having a carboxylic group at each end of the hydrocarbon chain. It is evident that adsorption of the di-carboxylic soap takes place through both polar groups as on a water surface; otherwise, if only one group were adsorbing, the other polar group sticking outwards would give a hydrophilic character to the coating, regardless of the length of the hydrocarbon chain. However, in the case of azelaate (C_9) the length of the chain is insufficient to give effective association with frother molecules, whereas the much longer hydrocarbon chain (C_{16}) of thapsate is sufficient to give stronger association with nonpolar groups of its own molecules, or of frother, to effect excellent flotation.

(d) Frothing and Flotation Tests. Procedure: Effects of additions of collectors on the froth-volume of frothers were studied in a frothing setup similar to one used by Sun.[18] Flotation tests were conducted in a 100-ml all-glass cell with a sintered disc partition, aerated by compressed nitrogen. Rate of aeration was measured by a flowmeter and was kept constant for a series of tests. Homogeneous solid particles, air atomized Cu powder, ground galena, sphalerite, and barytes, were used at 1:4 S:W (solid:water ratio).

Results of frothing tests are shown in Tables III and IV and, for straight-chain alcohols, in Fig. 7.

The froth of individual alcohols used as frothing agents is greatly affected by the addition of xanthates, the effect being shown by the variation of froth-volume and by the changes in the character of the resultant froth. The results show that when a constant amount of ethyl or amyl xanthate is added the froth volume at first decreases and then increases, with increasing chain length of alcohol, see Fig. 7. The detailed tracing of effects of frothing of mixtures is complicated by the simultaneous changes in the character of the froth. However, the order of magnitude of the effect of xanthate additions on the frothability of alcohols is similar to that of the increase of the surface pressure obtained in penetration experiments on the Langmuir trough, which are attributed to molecular interactions. This suggests

Table III. Effect of Collector (Xanthate) Addition onto Froth-Volume (Height of Froth in mm ± 5) of Frother*

Concentration of Frother	Initial Frother Volume	Final Froth Volumes with Collectors (40 ml soln) KEX	KAX	K Lauryl Xanthate
	20 ml	alone	alone	alone
—	—	10.1	15.f	20.F
M/1000 iso-butyl alcohol	20.f	10v.f	25v.f	nil
M/1000 2 methyl 2 butanol	35.m	30.m	65v.f	nil
M/1000 4 methyl 2 pentanol	70.l	75.m	80v.f	70.F
M/1000 2 ethyl hexanol	65.m	10.m	95v.f	90.F
M/1000 methyl monyl carbinol	20.1	5.m	25.f	nil
M/1000 phenol	15v.f	20.1	25.m	nil
M/1000 o-cresol	40.f	25.m	65v.f	nil
M/1000 m-cresol	45.f	30.m	50v.f	10.F
M/1000 p-cresol	45.f	35.m	70.f	nil
M/1000 cyclo-hexanol	45.m	35.f	50.f	nil
M/1000 o-methyl cyclo hexanol	55.m	60.f	85v.f	nil
M/1000 p-methyl cyclo hexanol	65.m	75.m	90 foam	nil
M/1000 α-Terpineol	55.m	85.m	95.m	60.F
M/1000 ($C_{12}H_{25}SO_4Na$) sodium dodecyl sulphate	200—350 l&m	350—400.m	400—500.m	140—170.F

* 20 ml frother and 20 ml of xanthate, both M/1000 solutions; const. aeration by nitrogen, temp. 18° to 20°C, pH neutral, time 5 to 10 min; diam of frothing cylinder 42 mm.
Character of froth:
v.f. very fine bubble froth
m. medium size, approximately ⅛ to ½ in. diam
l. large size, above ½ in. diam
f. fine bubbles, below approximately ⅛ in. diam
F. foam, air dispersed in solution

that molecular interactions are also operative in the frothing phenomena. The analogy is not complete, as in the frothing tests the experimenter is almost certainly dealing with non-condensed films; in Langmuir trough experiments the films are condensed.

Comparison of the effects of straight chain with branched chain and substituted cyclic alcohols, which are known to change the type of molecular association leading to less condensed films, indicates that condensation of interfacial films deters frothing.

The most interesting examples of the effects of stereochemistry and polarity of associating components on frothability are those given by systems dodecyl sulphate: cetyl trimethylammonium bromide (C.T.A.B.) and dodecyl sulphate—dodecylamine hydrochloride, Table IV. Although dodecyl sulphate and C.T.A.B. react strongly to give a 1:1 complex resulting in a formation of a visible precipitate, the size of the bulky polar group $N(CH_3)_3^+$ is such that the resultant $SO_4^-:N(CH_3)_3^+$ grouping of the complex possesses residual polarity which enables the complex to orientate itself preferentially and to spread at the air/water interface (instead of remaining as precipitate in solution) once aeration is started. This tendency leads to the formation of a strong, liquid condensed, non-solidified film, giving tenaceous, stable cellular froth. On the other hand, the interaction between $C_{12}H_{25}SO_4^-$ and $C_{12}H_{25}NH_3^+$, which leads also to a 1:1 complex, gives a visible solidified, strongly hydrophobic film at the interface, too brittle to withstand any vibration, and the solution is incapable of frothing.

Table IV. Examples of Frothing of Collector-Frother Reagents*

Reagents	Frothing	Remarks
$C_{12}H_{25}SO_4Na$	200 to 350 with large air "holes"	comparatively stable (2 to 5 min)
CTAB	400 to 500 with large air "holes"	comparatively stable
$C_{12}H_{52}SO_4Na$ + CTAB	>> 500 in spite of visible precipitation occurring on mixing the two reagents	"honey-comb" structure, stable froth of large size, practically without water
$C_{12}H_{25}SO_4Na + C_{12}H_{25}NH_3Cl$	nil	no precipitate but visible brittle film found on air-liquid interface
$C_{12}H_{25}SO_4Na + C_8H_{17}OH$	250 to 280, foamlike	comparatively stable
$C_{12}H_{25}SO_4Na + C_2—C_6OH$	350 to 500, foamlike	less stable than with $C_8H_{17}OH$
$C_{12}H_{25}SO_4Na$ + α Terpineol	300, foamlike	very stable (for more than 15 min)

* 20 ml of M/1000 solutions used, temp. 18° to 20°C, time 5 to 10 min, pH neutral; height of froth in mm ±5, diam of frothing cylinder 42 mm.

Characteristics of the froth required in flotation cannot yet be described in quantitative terms. The required froth is spoken of as copious, neither too brittle nor too tough. These requirements are in some way related to the type of film produced by molecular interactions between orientated molecules at the air/water interface.

Correct molecular associations between frother and collector molecules must be suitably balanced to give appropriate mechanical properties to the air/water interface.

Results of recoveries obtained by floating galena in a pneumatic cell, using equivalent amounts of frothers and collectors, are presented in Table V. These results show the same characteristic effect

Table V. Flotation Tests in Pneumatic Cell*

Frother	Recovery, Pct KEX	KAX	Remarks
M/100 C_3H_7OH	14.5	—	gradual increase in volume and stability of frothing and mineralization, giving an easily overflowing froth for C > 6
M/100 C_4H_9OH	32.0	—	
M/1000 $C_5H_{11}OH$	52.5	66.5	
M/1000 $C_6H_{13}OH$	65.0	74.5	
M/1000 $C_7H_{15}OH$	72.5	83.0	
M/1000 $C_8H_{17}OH$	76.0	85.5	
M/1000 4 methyl 2 pentanol	71.5	80.0	good froth, brittle
M/1000 2 ethyl hexanol	86.5	97.5	copious, good consistency froth
M/1000 methyl nonyl carbinol	51.0	37.5	good mineralization, tough foam
M/1000 o-cresol	55.0	86.5	poor frothing though good mineralization of bubbles
M/1000 m-cresol	64.0	84.5	
M/1000 p-cresol	69.0	82.0	
M/1000 α Terpineol	88.5	98.0	abundant, good consistency frothing

* Using 20 g galena −60 +180 mesh, 60 ml distilled water, 10 ml frother solution, and 10 ml M/1000 xanthate solution. pH 7.5, 1 min conditioning time without aeration, 3 min flotation time using compressed N_2 for aeration.

of progressively increasing molecular associations xanthate-alcohols, leading to higher recoveries, which subsequently drop off when associations are too strong and condensed brittle films incapable of withstanding vibrations are formed. Suitable choice

of branched chains and/or substitution of CH_3 and OH groups in cyclic chains are extremely effective in giving better recoveries. The effects on frothing of frother-xanthate mixtures are, however, not exactly parallel to the flotation recoveries obtained by these mixtures. It is evident, therefore, that the solid itself also affects the mineralized froth.

Cu powder, well-floating at pH 5.5 to 9.0, with dodecyl sulphate alone still gives a very good flotation if an equivalent amount of C.T.A.B. is added to the cell, though the resultant froth is much more stable and too voluminous for commercial purposes. However, if an equivalent amount of dodecyl amine hydrochloride is added to the solution of dodecyl sulphate floating Cu, the flotation is completely suppressed. This was found to be due not only to the suppression of frothing itself, but also to the actual desorption of dodecyl sulphate from Cu surface by the complexing action of amine. Residual Cu powder is completely hydrophilic and free to adsorb another collector, like xanthate or even excess of dodecyl sulphate, if added above the equivalent amount of amine hydrochloride. Presence of strong alkyl sulphate—alkyl amine complex in the solution is, however, hindering to efficient adsorption and froth formation of excess dodecyl sulphate (and xanthate plus frother) and prolonged conditioning seems to be required to overcome the interference of the complex in solution. If the harmful complex is decanted, the residual Cu powder reacts with further added collector and frother at a much higher rate and flotation takes place almost immediately.

The concept of molecular interactions between collector and frother molecules makes it possible to visualize the process or mechanism involved in dynamic attachment of air bubbles to solid particles. Fig. 8a shows the distribution of collector and frother molecules at interfaces and in bulk solution at optimal flotation conditions, as an air bubble and a particle approach each other. Both at the air/water interface and at the solid/water interface *diffuse monolayers* of associated and unassociated collector and frother molecules are present, temporarily in equilibrium with molecules in bulk solution. The film at the solid surface differentiates from that at the air/water interface insofar as the collector molecules are irreversibly adsorbed (under given flotation conditions) and the frother molecules are free to adsorb and desorb. As soon as the air bubble contacts the solid surface, Fig. 8b, the collector-frother molecules of the air/water interface can now penetrate the diffuse monolayer at the solid, and adsorb strongly onto the solid surface, greatly increasing *local* surface concentration and *local hydrophobic* character of the surface. At the moment of attachment of a particle to an air bubble the mixed monolayer at the air/water interface becomes slightly more condensed and consequently the bubble is stabilized. A well-mineralized bubble constitutes a more stable froth system.

If the films at the air/water and the solid/water interfaces, Fig. 8a, are too highly condensed, the adherence of the bubble is temporarily prevented, since a stabilized hydrophilic surface of the air bubble is approaching a stabilized hydrophobic surface of the solid, giving initial conditions for the formation of a bimolecular-leaflet micelle, the so-called armoring of bubbles. These conditions are achieved more readily by strongly associating collector and frother molecules, although the concentration of collector may be still far from the critical

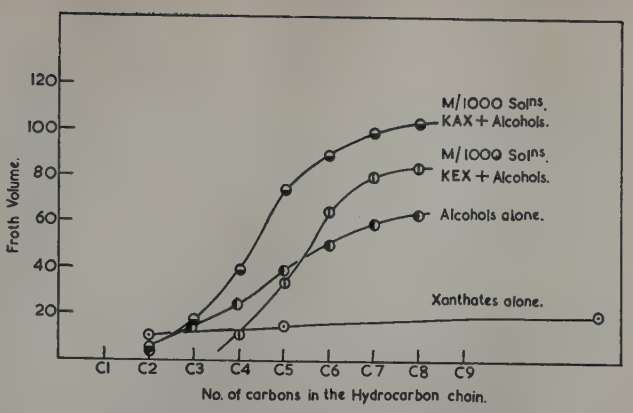


Fig. 7—Effect of xanthate addition on frothability of straight-chain alcohols. Molecular associations between collector and frother, in relation to froth volume.

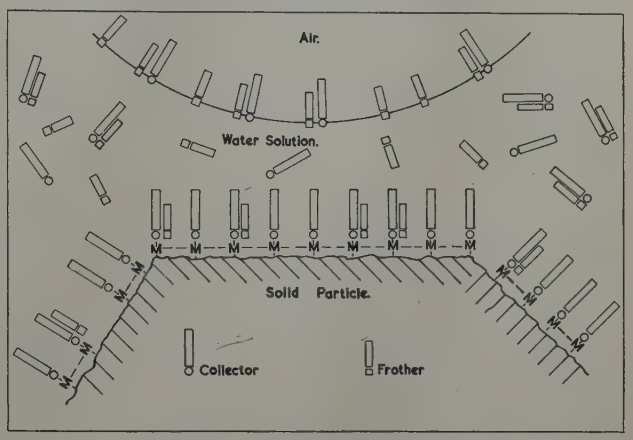


Fig. 8a—Mechanism of bubble attachment: bubble approaching a collector-coated solid surface; diffused monolayers of associated and unassociated molecules at interfaces and in solution.

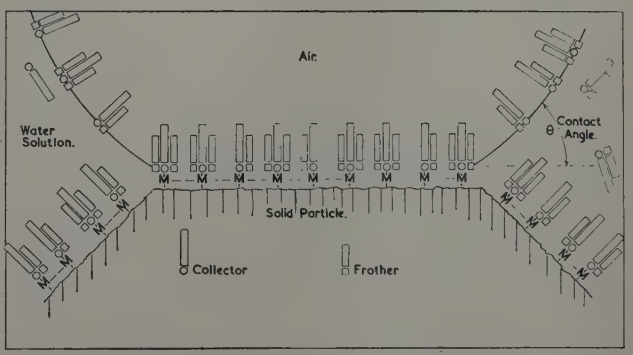


Fig. 8b—Mechanism of bubble attachment: adherence of an air bubble established through the penetration of the monolayer at the solid/liquid interface by the monolayer at the air/liquid interface.

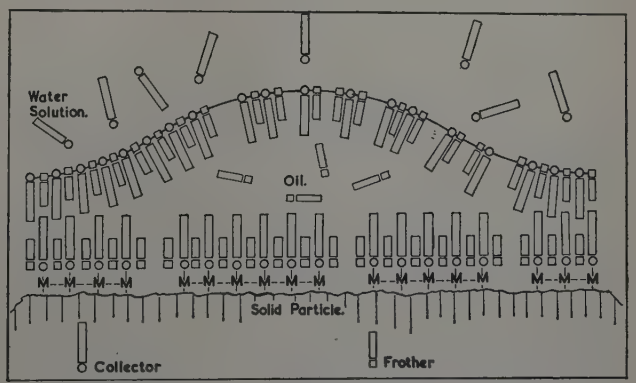


Fig. 9—Mechanism of oil-spreading on a highly hydrophobic solid surface.

concentration for micelle formation. It can be readily seen that with a fully condensed monolayer of the collector immobilized on the solid surface, no penetration by the frother molecules can be obtained, in contrast to the case with frother/collector penetration at the air/water interface, where the monolayer molecules are mobile. The impossibility of penetration prevents bubble attachment.

Gaudin et al.,[18a, b, c] Simard et al.,[19] and Schulman and Smith[20] have shown that without exception optimum flotation recoveries are obtainable with a fraction of a collector monolayer coverage, e.g., with diffusely covered surfaces. Using radioactive isotopes, Beischer[21] has demonstrated experimentally that the molecules adsorb primarily on corners and edges of scratches, where the surface energy is minimum and the adsorption energy maximum.

The strongly hydrophobic nature of densely collector-covered surfaces which prevents penetration by frother molecules causes further (a) adherence of particles to one another (flocculation, due to van der Waals forces) and (b) spreading of oils (derived from flotation reagents and from mining operations) and of the degradation products, such as dixanthogens. In the case of oil-spreading flotation is impaired owing to engulfment of bubbles by the oil films (overoiling phenomenon), which decreases the firmness of bubble adherence. The spread oil films also dissolve the frother molecules and remove them from the flotation pulp. Fine particles, possessing many highly reactive edges and corners, are more susceptible to overoiling and this is an additional cause for their non-floatability, besides the aging of surfaces, as described by Gaudin et al.[22]

Fig. 9 shows how contact angles greater than 90° can be easily obtained by replacement of air by oil. The much more readily stabilized monolayer at the oil/water interface can form a bimolecular leaflet with the collector-frother coated surface, giving higher angles which are impossible to attain at the air-water. This is readily explained by the fact that the surface tensions at the air/water ($\gamma a/w$) interface are rarely lowered by surface active agents to less than 30 dynes per cm, whereas the surface tensions of less than 1 dyne per cm can be easily obtained at the oil/water interface, $\gamma o/w$.

The following relationship holds:

$$\frac{\gamma s/o - \gamma s/w}{\gamma o/w} = \text{Cos}\ \theta$$

Waterhouse and Schulman[23] have shown by electron-diffraction pattern that the diffusely collector-covered solid surface is penetrated by frother molecules, giving a new diffraction pattern of the mixed monolayer.

Summary

The experiments carried out with a variety of pure surface active agents, employed in flotation of ores as either collectors or frothers, show that (a) long-chain homologues of alcohols and alkyl sulphates react strongly with xanthates at the air/liquid interface (Langmuir-trough experiments); (b) the reactions between frothers and collectors occur at the solid/liquid interface, if the solid is suitably coated by collector molecules (frother abstraction tests); and (c) the effects of molecular associations collector-frother are evident in frothing and flotation results. The measurements of air/water/solid and oil/water/solid contact angles (carried out under variable conditions as regards concentration of the surface active agents, pH, time of conditioning, and presence of complexing agents in the oil phase) reveal the importance of the density of collector-coverage. A mechanism for attachment of suitably collector-coated particles to an air bubble is postulated.

Acknowledgments

The authors wish to thank the following companies for supplying some flotation reagents and chemicals: the American Cyanamid Co., the Hercules Powder Co., and the Imperial Smelting Corp. for very good specimens of minerals.

References

[1a] S.-C. Sun: *Trans.* AIME (1952) **193**, pp. 65-71.
[1b] S.-C. Sun: *Trans.* Institute of Mining and Metallurgy (April 1952).
[2] J. H. Christman: American Cyanamid Co. (1930) *T.P.* 17.
[3] W. Wark: *Principles of Flotation.* Australasian Institute of Mining and Metallurgy (1938).
[4] A. F. Taggart and M. D. Hassialis: *Trans.* AIME (1946) **169**, p. 259.
[5] S. A. Wrobel: Flotation Frothers, Symposium on Mineral Dressing. Institute of Mining and Metallurgy (London 1952).
[6] A. F. Taggart: *Handbook of Mineral Dressing,* Section 12. John Wiley and Sons, New York, 1947.
[7] S. R. B. Cooke: *Advances in Colloid Science,* III, 1950, *Flotation,* p. 326.
[8] N. K. Adam: *Physics and Chemistry of Surfaces.* Oxford University Press, 1941.
[9] J. H. Schulman and A. H. Hughes: *Journal of Biochemistry* (1935) **29**, pp. 1236, 1243.
[10a] J. H. Schulman and E. K. Rideal: *Proc.* Royal Society B 122 (1937) p. 29; *Nature* (1939) **144**, p. 100.
[10b] J. Marsden and J. H. Schulman: *Trans.* Faraday Society (1938) **34**, p. 748; J. H. Schulman and E. Stenhagen, *Proc.* Royal Society B 126 (1938) p. 356.
[10c] E. G. Cockbain and J. H. Schulman: *Trans.* Faraday Society (1939) **35**, p. 1.
[10d] R. Matalon and J. H. Schulman: *Trans.* Faraday Society (1947) **43**, p. 479.
[10e] E. D. Goddard and J. H. Schulman: *Journal of Colloid Science* (1953) **8**, p. 309.
[11a, b, c] G. A. Wolstenholme and J. H. Schulman: *Trans.* Faraday Society (1950) **46**, p. 475; (1950) **46**, p. 488; (1951) **47**, p. 788.
[12] J. G. N. Thomas and J. H. Schulman: To be published.
[13] F. D. Snell and C. T. Snell: *Colorimetric Methods of Analysis,* D. van Nostrand Co., 1937.
[14] Hartley and Runnicles: *Proc.* Royal Society A (1938).
[15] S. R. Epton: *Trans.* Faraday Society (1948) **44**, p. 226.
[16] B. D. Cuming and J. H. Schulman: Private communication (see Schulman and Leja, Kolloid Zeitschrift Symposium, 1953).
[17] B. D. Cuming and J. H. Schulman: Recent Development in Mineral Dressing, Symposium of the Institute of Mining and Metallurgy (London, 1953).
[18a] A. M. Gaudin and C. S. Chang: *Trans.* AIME (February 1952) p. 193.
[18b] A. M. Gaudin and W. D. Charles: *Trans.* AIME (February 1953) p. 195.
[18c] A. M. Gaudin and F. W. Bloecher: *Trans.* AIME (February 1950).
[19] C. M. Judson, A. A. Lerew, J. S. Kennedy, R. B. Booth and G. L. Simard: *Trans.* AIME (April 1952) p. 375.
[20] J. H. Schulman and T. D. Smith: Recent Development in Mineral Dressing, Symposium of the Institute of Mining and Metallurgy (London, 1953).
[21] D. E. Beischer: *Journal of Physical Chemistry* (1953) **57**, p. 137, and previous work.
[22] A. M. Gaudin, et al.: *Trans.* AIME, **112**, p. 303.
[23] B. Waterhouse and J. H. Schulman: To be published.

Upgrading Manganese Ore

Three Kids Mine, Nevada

by S. J. McCarroll

Fig. 1—The belt shown at right carries filter cake to mixing station over calciner. Crude ore conveyors appear in right background.

THE Three Kids mine, some six miles east of Henderson, Nev., is in a typical southwest desert area, with high dry summer heat and cool to cold winter seasons.

The manganese deposit was located during World War I.[1] During this period 15,000 to 20,000 tons of ore assaying up to 41 pct manganese were shipped. Interest in the deposit was not revived until the middle thirties, when experiments on the ore were initiated. Test work indicated possible recovery of only 70 pct by flotation, but in 1941 additional work was done at the Boulder City pilot plant of the U. S. Bureau of Mines and also by M. A. Hanna Co. As a result, the Manganese Ore Co. was formed and a plant utilizing the SO_2 process was constructed. Numerous operation difficulties ensued, and the plant was closed when the manganese situation in the country eased.

In 1949 Hewitt S. West initiated negotiations to acquire the plant. In 1951 Manganese, Inc., was formed and contract entered into with the General Services Administration to supply 27 million units of metallurgical grade manganese in the form of nodules to the national stockpile. A second contract was made to upgrade 285,000 tons of stockpile ore.

Test work was undertaken by the Southwestern Engineering Co. and likewise by the Boulder City pilot plant at the U. S. Bureau of Mines. Results obtained indicated the commercial feasibility of the flotation process. Construction of the plant, which is shown in Figs. 1 and 2, was started in June 1951, and operations on a break-in basis began in September 1952. Apart from the usual starting difficulties two major disasters caused serious setbacks, one a kiln failure in February 1953, and the other a fire that destroyed the flotation building in June of the same year. The nodulizing section of the plant resumed operation in November, and the flotation section in January 1954.

The ore minerals are chiefly wad,[2] with minor amounts of psilomelane, and occur in sedimentary beds of volcanic tuff. The ore is overlain with beds of gypsum which outcrop or may be covered with surface gravel. Intermediate beds of red and white tuff occur frequently with lenses of red and green jasper and stringers of gypsum and calcite. Small amounts of iron are present; lead content averages about 1.0 pct and minute amounts of copper and zinc are found. Barite, celestite, and bentonite are present. Since these are made up of minute asicular crystals, moisture content is very high, averaging about 18 pct. Ore reserves have been estimated at 3 million tons averaging 18 pct Mn[2] and up to 5 million tons after grade is dropped to 10 pct Mn.

A good part of the orebody was stripped of overburden by the previous operating company. Approximately 50 pct of the ore, representing more than 60 pct of the manganese, can be mined by open-cut methods. A system for underground mining has not yet been decided on.

Open-cut mining with benches of 20 ft has proved satisfactory. Although the ore is soft and appears dry and dusty it has a certain resilience, probably due to the porosity and moisture which makes drilling and fragmentation difficult. Wagon drills have been abandoned in favor of the Joy 225-A rotary drill which will put down a 4¾-in. hole at the rate of 2 ft per min. Holes are spaced in a pattern with 8 to 9-ft centers. Forty percent powder has been used, but better breaking to 2-ft size is obtained with low velocity bag powder of 30 pct strength.

Loading is done with one 2½-yd shovel, and cleanup follows with one D-7 bulldozer. The ore is hauled with Euclid trucks about 1000 ft from the pit to a blending pile, where the daily mine production is spread in layers by bulldozing until approximately one month's mill feed is accumulated. A new pile is then started and mill feed is drawn from the first pile by one 1¾-yd shovel and Euclid trucks, with a haul of approximately 500 ft.

Mining is performed by an independent contractor with engineering and supervision by the company staff.

Early test work indicated that the manganese could be floated with soap, a wetting agent, and fuel oil to give a recovery of better than 75 pct with a grade of 43 pct Mn. The concentrate when nodulized with coke would upgrade to 46 pct Mn or over, and the lead volatilized to 0.6 pct residual.

S. J. McCARROLL, Member AIME, is General Superintendent, Manganese, Inc., Henderson, Nev.

Discussion on this paper, TP 3747B, may be sent (2 copies) to AIME before May 31, 1954. Manuscript, Sept. 8, 1953. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 228. The intervening non-Transactions pages appeared in Mining Engineering.

Fig. 2—Flotation mill and kiln plant, ore blending piles in background.

Experimental and operating results show the concentrate to lose from 12 to 15 pct by weight in the calcining and nodulizing process, owing to volatilization of CO_2, SO_2, and water of crystallization. An increase in manganese percentage is apparent and is depended upon in plant operation. No trouble is encountered when recoveries are calculated on a unit basis from the heads to the final nodule product over a period of time, because both products are weighed. The loss in weight does present problems in test work and analysis which will be discussed after the test procedures have been outlined.

The metallurgical data in Table I illustrate pilot plant results for one test. Upon nodulizing, the concentrate would show 15 pct insoluble, the upper limit on metallurgical grade nodules.

Table I. Pilot Plant Metallurgical Results, July 5, 1950

Product	Wt, Pct	Mn, Pct	SiO_2, Pct	Al_2O_3, Pct	Distribution
Dry ore	100.0	25.55			100
Rough conc't	64.9	36.36			
Cleaner conc't No. 1	58.1	39.08			
Cleaner conc't No. 2		39.59			
Cleaner conc't No. 3	50.2	42.30	10.67	2.64	83.1
Rougher tail	35.1	5.55			7.6
Cleaner tail No. 1	6.8	13.32			3.5
Cleaner tail No. 2	7.9	18.98			5.8
Cleaner tail No. 3	Recirculated				
		Ratio of Concentrate			1.98:1
Reagents Used		**Lb per Ton**			
Fuel oil		65.6			
Wetting agent		6.0			
Manganese sulphate (65 pct)		26.0			
Vegetable oil soap dry		47.6			
Sodium silicate		0.5			

Feed rate 1000 lb per hour. Lake Mead water used.
All reagents added separately to classifier overflow except 21.4 pct of total soap, which was added to No. 1 conditioner.

After completion of pilot plant flotation and nodulizing tests, work continued on reagent combinations and quantities in an attempt to make satisfactory recoveries on lower-grade ore and obtain a lower insoluble in the concentrate. Straight run and cracked oils were investigated as well as the effect of pulp temperatures.

An increase in the amount of fuel oil aided materially in lowering the insoluble. With the oil increased to 120 lb per ton, the dispersed condition of the concentrate disappeared. The concentrates assumed a granular form as the slimes coagulated, and the combined insoluble dropped to 8 to 10 pct. The oil emulsion method of manganese flotation has proved more stable and easier to control in plant operation than the dispersed method. Recent U. S. Bureau of Mines work[3] arrives at the same conclusions.

Soap skimmings, a byproduct of the sulphate paper industry, was not only the cheapest collector but gave equally good recovery and was available in the quantities required. The test work further indicated that an emulsion of oil, soap, and oronite-S added at the conditioning stage seemed to give better results than stage additions, so this procedure was adopted. Liquid SO_2 was added in a 3 pct water solution just ahead of the conditioning stage. The Ph value for conditioning and floating the ore is not critical and generally regulates itself between 7.8 and 8.6 pct, and total reagent consumption per ton of ore has averaged between 250 and 300 lb.

The elimination of reagents from samples to determine the exact manganese content has posed a problem, which as yet has not been solved in a satisfactory manner. Solvents do not easily remove the insoluble metallic soaps and oil by any sampling method rigid enough for test work or mill samples. The method now used is to dry the sample slowly, igniting the filter paper while the sample is still hot. The sample generally ignites itself and is partly calcined. The effect on the manganese content on laboratory samples is not serious, but wide variations did occur on the mill samples prepared in this manner. High assays occur, especially when unusually large amounts of reagents have been used, or when the ore being treated contains much of the sulphate minerals. Determination of intermediate mill products is therefore inaccurate; consequently the results of milling and nodulizing operations must be calculated on the basis of nodule units produced against crude ore units delivered to the mill.

Table II. High Oil Emulsion Laboratory Test No. 28, February 1952

Product	Wt, Pct	Mn, Pct	SiO_2, Pct	Al_2O_3, Pct	Pb, Pct	Distribution
Heads		21.31				
Redried concentrate	40.5	45.5	6.7	2.5		84.0
Combined middlings	8.4	12.1				4.6
Tails	39.9	4.7				8.4
Nodules	37.2	49.5	7.3	2.6	0.61	84.0
Reagents Used			**Lb per Ton**			
Dry St. Marys soap skimmings			40			
No. 1 fuel oil			120			
Oronite S wetting agent			6			
SO_2 as 5 pct sol.			8.5			

Conditioning 5 min, rough 3 min, clean four times. Conditioning at approximately 15 pct solids, flotation at 10 pct solids.

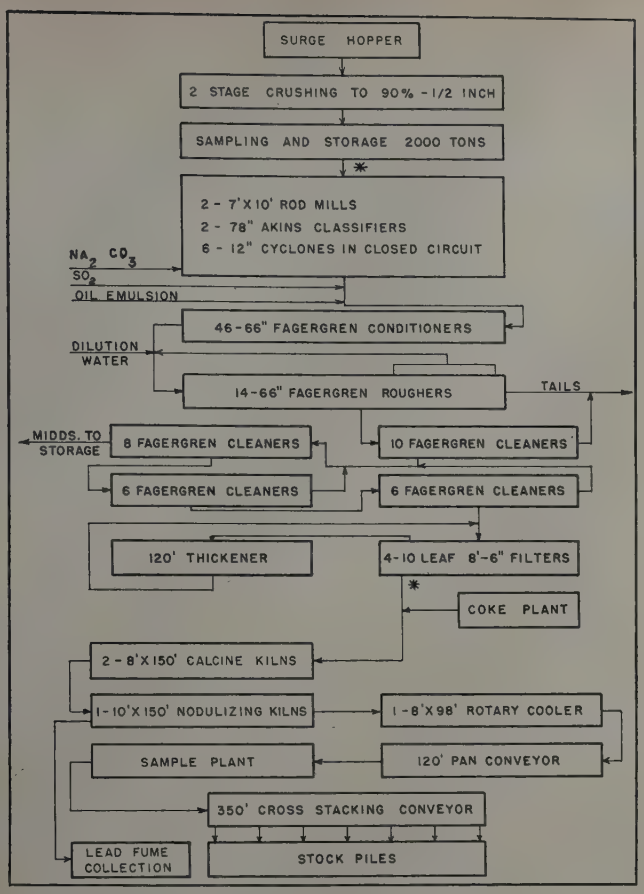


Fig. 3—Simplified flowsheet of major equipment and process steps at Three Kids mine.

A typical laboratory test using the high oil emulsion is shown in Table II. The reagents here amount to 166 lb per ton, and as nearly all this remains, reagents account for over 15 pct of final concentrate.

Conditioning in the laboratory is carried on in a Fagergren test cell for 5 to 10 min at densities of 15 to 30 pct solids. Although thousands of tests have been run in the laboratory at Manganese, Inc., the proper combination of power, motion, time, and air is still undetermined. The failure to correlate mill results with laboratory test work has led to many experimental changes in the mill conditioning. Even though improvements in conditioning have been made, the consumption of reagents in the mill is still greater than laboratory tests indicate they should be.

The plant is designed to treat 1200 long dry tons of ore containing 18 pct moisture. An area of more than 20 acres is required for the ore bins, crushing plant, flotation section, nodulizing kilns, conveyors, thickeners, shops, offices, and electrical substations. To conserve space a simplified flowsheet of the major equipment and process steps is shown in Fig. 3, and only those points which present unique problems will be discussed. Power is supplied from the lines of the Basic Management, Inc., at 69 kv to the main substation where the voltage is dropped to 2300 for distribution to three substations supplying the crushing plant, flotation plant, and kiln section. The primary crusher and rod mill motors operate on 2300 v, while the rest of the plant uses 440-v current.

Water is supplied from the 42-in. line of Basic Management, Inc., which crosses the property.

In the crushing section 5-in. discharge from the primary jaw crusher is scalped and the $+\frac{1}{2}$ in. is fed to one 30x30 double impact breaker where a product of 90 pct $-\frac{1}{2}$ in. is obtained in open circuit. Previous attempts to use cone-type crushers were unsuccessful, because of the pancake effect of squeezing the moist spongy ore. The tuffaceous ore is not too abrasive for the impact-type crusher.

Even though the ore contains 18 pct moisture when delivered from the mine, or about 12 pct from the stockpile, both crushing operations produce considerable dust. This problem is adequately taken care of by a Sly dust collector system.

Table III indicates the size of secondary crusher product. The percentage of fines has been higher than anticipated; therefore the plant now in construction employs $\frac{1}{4}$-in. rod deck screens to send $+\frac{1}{4}$ in. directly to the mills and the undersize to the spiral classifier, from which the 10 mesh and finer material will overflow to cyclone classification, Fig. 4, where a 65 mesh separation is made. The +65 mesh returns to the mill for regrind.

Because of bentonite in the ore mechanical classification was impossible, even though the classifiers were lowered to a point where the weir and return discharge were at the same level and the pulp diluted to 12 pct solids. The introduction of cyclones in closed circuit with the mills and spirals has been the solution to a baffling problem.

Soda ash is fed in amounts of 5 to 20 lb per ton before the cyclones. Part of the gypsum dissolved in the grinding is thus precipitated and the tendency to form insoluble calcium soap after addition of the emulsion is reduced.

It is believed that when a relation is established between power, motion, dilution, and particle size the uncertainties of oxide manganese flotation will have been overcome.

Table III. Noncumulative Screen Sizing, Pct

Material	+¼	−¼	−10	−65	+65	+100	+200	−200
Impact crusher discharge	23	77	57	23				
Spiral class overflow					6.7	9.5	15.3	68.5
Cyclone overflow					0.4	2.1	7.5	90.0

On the basis of laboratory and pilot plant test work, 160 hp was the connected load for conditioning. The power and number of conditioners was gradually increased to 690 hp, at which time a maximum of 850 tons per day was treated. Sixteen additional cells using 480 hp were in the process of installation at the time of the fire. From past results

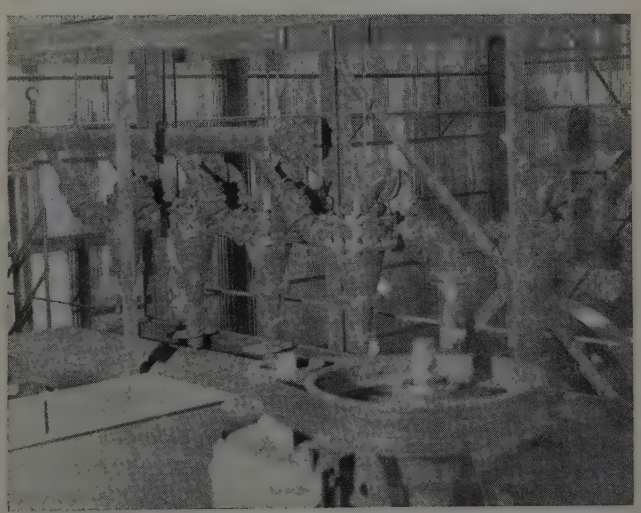


Fig. 4—The 12-in. cyclones shown above are used as classifiers in the grinding circuit.

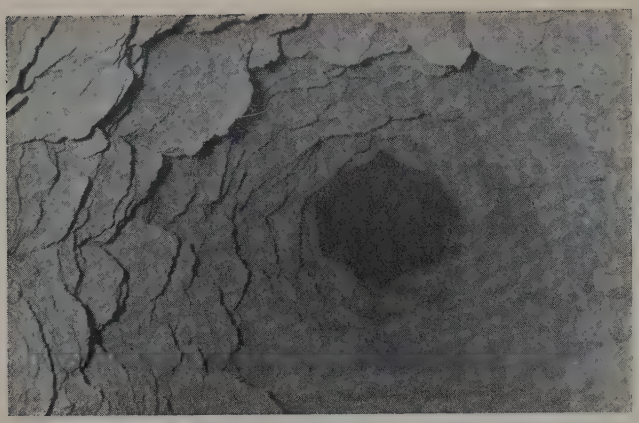


Fig. 5—Interior of nodulizing kiln showing buildup of manganese on brickwork.

it is believed that the total of 1170 hp being installed in the new plant will give the conditioning necessary to bring the plant to full production.

Among other variables experimented with in the mill conditioners were corner and side baffles, underfeeding, low and high pulp levels, several types of impellers, and the introduction of small amounts of air and stage addition of reagents.

Fagergren rotors operating with a peripheral speed of 2250 fpm in 66-in. cells have been found to be the most effective conditioners. Perforated baffles placed at right angles to the sides of the tanks give the best results. The pulp level must be several inches over the rotors. Encouraging results have been obtained using stators around the rotors, with all air sealed off. Fourteen cells thus equipped are in use. The power used is only half that of the straight rotor cells, but the pulp level must be low and the retention time is reduced to a point where total efficiency is about the same as the rotor cells with double horsepower.

Conditioning time provided is 13 to 16 min, depending on exact pulp level and percent solids. Little difference in conditioning has been found in the range of 15 to 28 pct solids. Laboratory work has not indicated better conditioning with thicker pulp, difficult because of classifier limitations.

Effectiveness of conditioning is determined visually in the mill by dip samples taken with a beaker. If conditioning is complete the mineral will rise in a wide band and the gangue will settle on the bottom with a clear band of water between the two. Unless the band of water appears it is almost certain that the pulp is not conditioned enough for flotation.

Each flotation circuit uses seven 66-in. cells for roughing and four stages of cleaning. Some products are recirculated to supply water and stability to the circuit.

The conditioned pulp is diluted to 10 pct solids as it enters the roughers. Tailings are discharged at 5 to 6 pct solids. Dilution is essential in the cleaning. Feed and concentrate average 12 pct solids with tails showing 2.5, 1.7, 1.0, and 0.5 pct solids as cleaning progresses. A middling which amounts to 3.5 to 5 pct of the Mn in the ore is bled off and stored for future treatment.

With good conditioning the froth is a heavy matte about 2 in. thick and the bubbles have a bright appearance. An excess of reagents will flatten the froth to a scum, while a lack of reagents creates great volumes of froth without mineral flotation. The emulsion is fed by metering pumps with remote controls and rate indicators on the operating floor.

The Cattermole effect, so troublesome in Cuba,[4] has caused no difficulty in launders, although buildup is troublesome on the flotation cell stators. The oily concentrate causes all rubber and neoprene compounds to swell, and all such parts in contact with the pulp have been replaced with steel.

It has not been possible to operate a thickener on the mill concentrates. The repeated handling or flowing of the concentrate causes the heavy flocks to coagulate into beads or even balls, which stop up pumps and lines. Filters have been operated successfully by feeding the mill concentrate at 12 pct solids directly to the filter tanks. A certain amount of settling takes place and stirring of the tanks is necessary. When concentrate is fed directly to the filters there is a constant overflow of slimes which is thickened and returned to the filters.

Eight 12-in. cyclones have been installed above the filters to act as thickeners. The slime overflow is thickened in one 120-ft traction thickener, where a certain amount of coagulation takes place and then returns to the filters through the cyclones. Drags and classifiers similar to those used in Cuba[4] were considered for partial dewatering, but the variation in concentrate consistency from granular to dispersed indicated that poor separation would result. Cyclone tests indicate that even with highly dispersed pulp some thickening takes place. It is believed that the cyclone installation will increase the filter feed solids from 12 to 25 pct and thereby double the filtering capacity. Filter cake is voluminous and contains 30 to 35 pct moisture.

A small crushing and screening plant reduces stockpile petroleum coke to —⅜ in., which is fed in amounts of 3 to 5 pct of the dry concentrate weight to the filter cake conveyor.

The flow of wet filter cake and coke is diverted at 15-sec intervals between two 8x150-ft rotary kilns. Two pug mills were used to mix the coke and filter cake thoroughly. The discharge was fed through inclined pipes to the kilns. Stopping up of the feed pipes and backfeeding or spillage into the hood became so troublesome that ribbon-type screw conveyors are now being installed to do the job of mixing and feeding.

The wet filter cake is self-calcining after the kilns have been brought to 800°F by oil firing at the discharge end. Feed end temperatures range from 750° to 1200°F depending on cake moisture. Calcine discharges at 800° to 1000°F through alloy pipes to the nodulizing kiln.

As the hot calcine passes down the nodulizing kiln the temperature is raised to a maximum of 2400°F at a point 20 ft from the discharge end. From that point to the nose cold air chills the nodules at 2000°F or lower. About midway in the kiln the temperature reaches 1650°F and lead begins to volatilize. It is believed that the coke reduces the lead minerals to globules of metal which are then volatilized as one of the oxides. Simultaneously, part of the coke reacts with gypsum present to liberate SO_2, which in turn combines with the lead oxide fumes to form lead sulphate. This assumption is based on operating results. When a deficiency of coke or of air occurs in the kiln the lead content of the nodules immediately rises. When nodulizer feed is low in gypsum the lead in the fume occurs in nearly equal parts as oxide and sulphate. As the gypsum in the feed increases the proportion of lead as sulphate exceeds 90 pct and free SO_2 becomes a problem in the scrubbing system.

The combined insoluble also appears to have some control over lead liberation. When the insoluble approaches or exceeds 15 pct the lead is volatilized with difficulty. This effect may be due to the exclusion of air by the more fluid nature of the tumbling charge or to the chemical combination of lead compounds as silicates.

While 10 to 12 pct insoluble is desirable to make good nodules, more than that amount causes ringing and build-up in the kiln, Fig 5, which must be shot or bored out. On several occasions the kiln has been cooled down and the manganese coating removed with pavement breakers. Alternate raising and lowering of kiln temperature has been found effective in causing the coating to drop loose. The exothermic reactions of the coke midway in the nodulizer contribute to the ringing, as frequently the temperature at that point will be higher than at the expanded nodulizing zone near the firing end. Although other plants[4-6] have discovered that an expanded zone is of benefit as a preheater in nodulizing, it is found in this instance to be detrimental. The lack of control over temperatures at the kiln center results in pasty material sticking in the expanded section until the walls are the same diameter as the rest of the kiln. In other words, preheating begins with the exothermic reactions, and nodulization in this kiln begins near the center and continues for nearly 75 ft.

The hot and often sticky nodules drop through the firing hood to a rotary cooler. Air enters around the nodulizer nose and is drawn concurrently through the cooler by an induced draft fan. Spray water is introduced in the nodulizer-cooler hood as a cooling aid. Pan and belt conveyors carry the cooled nodules through a sampling plant to the nodule storage piles, shown in Fig. 6.

Dust from miscellaneous points around the kilns is collected and returned by air slide to the calcining kilns. The stack gases of each calcine kiln pass through a wet cyclonic scrubber and the resulting manganese slurry is recycled through the mill concentrate thickener and filter plant. This dust has averaged more than 100 tons per day, but the use of screw feeders on the calciners is expected to reduce the load appreciably.

Small amounts of lead fume precipitate in the nodulizer multiclone dust collector, but it has not been determined whether or not returned dust contributes to high lead in the nodules. From the nodulizer multiclone gases pass to wet cyclonic scrubbers, where the major part of the lead fumes is collected. A Pease Anthony venturi-type scrubber was very effective in precipitating the lead fume, but mechanical problems due to SO_2 corrosion and gas volumes larger than anticipated necessitated complete revision of the fume collection system.

Fig. 6—A view of storage piles and stacker boom.

The system now installed uses one 70,000-cfm hot fan and two refractory-lined wet cyclonic scrubbers of 12 ft 6 in. and 9 ft 6 in. diam in series. An increase in gas velocity through the multiclone by addition of fresh air is expected to remove most of the dust now lost in the lead sludge as indicated in Table IV.

Table IV. Extracts from Metallurgical Statement, 15 Days of June 1953

Product	Short Tons	Mn, Pct	SiO_2, Pct	Al_2O_3, Pct	Pb, Pct	Long Ton Units	Recovery, Pct
Heads	10,116	24.3	28.48	5.65	1.16	219,526	100.00
Middlings	1,076	11.2				10,802	4.94
Tails	4,360	9.3				36,372	16.46
Conc't	4,678	41.3	9.58	2.50	1.60	172,352	78.60
Nodules	3,699	48.3	11.71	2.43	0.67	161,362	73.50
Dust loss						10,990	5.10

Reagents	32 Plus Diesel Oil	63 Pct Soap	42 Pct Oronite S	SO_2	Na_2CO_3
Lb per ton	189.9	100.4	9.7	8.2	11.6
Total 15 days	1,921,359	1,015,225	98,221	83,360	117,600
Nodulizer fuel, Bunker C oil			363,827 lb	83.77 lb per ton	
Petroleum coke			382,894 lb	4.0890 of conc't	

Adequate storage facilities for reagents are on the property. Emulsion is prepared in a special plant, each ingredient being fed in the right quantity through metering pumps equipped with variable controls at a central panel. A 50/50 mixture of reagents with water is first made, diluted with water to a 20 pct strength, and pumped to the 50,000-gal mill storage tank.

Conclusion

Manganese oxide flotation is still very much an art. The flowsheet and operating techniques will continue to change. Oxide ores of 18 pct Mn can be treated commercially. It is believed that studies of the physical properties of the amorphous and microscopic crystalline oxides may indicate a solution to effective conditioning and thus lower the cutoff point on ores now amenable to flotation.

Acknowledgment

Since the project began, Hewitt S. West, President of Manganese, Inc., has directed the process development, which is based on the work of Robert Lord and variations developed by Frank Trotter and the Manganese, Inc., staff. Suggestions made by the staffs of the U. S. Bureau of Mines at Boulder City and Salt Lake City in developing the process contributed materially to its success. Identification of bentonite by Edwin De Moss, Mine Superintendent, and John Atkins, Staff Engineer, opened the way for improved classification.

The author wishes to thank F. A. McGonigle of Manganese, Inc., for review of the paper and permission to publish these data.

References

[1] F. A. Hale: Manganese Deposits of Clark County, Nevada. *Engineering and Mining Journal* (1918) **105**, pp. 775-777.

[2] V. E. McKelvey, J. H. Wiese, and V. H. Johnson: Preliminary Report on the Bedded Manganese of the Lake Mead Region Nevada and Arizona, U. S. Geol. Survey *Bull. 948-D* (1949).

[3] Joe E. Rosenbaum and Carl H. Schack: Recent Improvements in Flotation of Manganese Ores. *The Mines Magazine* (March 1953) pp. 67-69.

[4] F. S. Norcross, Jr.: Development of the Low-Grade Manganese Ores of Cuba. *Metal Mining Milling Methods*, AIME (1943) p. 105.

[5] Anaconda Copper Co., Anaconda, Mont.

[6] Pioche Manganese Co., Pioche, Nev.

Manufacture of Tungsten Carbide Tipped Drill Steel

by T. A. O'Hara

SINCE May 1948, when tungsten carbide bits were introduced at the Flin Flon mine, they have been popular with the miners because of their fast drilling speed and low gage loss. The high cost of commercial carbide bits and tipped drill steel, however, prevented their use except for the hardest rock.

In an effort to extend the use of tungsten carbide on a basis economically competitive with detachable steel bits, experimental work was begun in 1950 to test the feasibility of making tungsten carbide tipped drill steel in the mine drill steel shop. This work showed that tipped drill steel could be made locally at less than half the cost of the commercial product. The performance of the local tipped drill steel was comparable to that obtained with commercial carbide bits and tipped drill steel and the cost per foot drilled was much lower.

Local tipped drill steel was adopted for all mine drilling in November 1951. Since then drilling costs per foot have been sharply reduced and footage drilled per manshift has increased markedly.

Experience at Flin Flon has shown that production of satisfactory carbide tipped drill steel is not difficult and that highly skilled labor and costly equipment are not required. As long as wise selection of brazing materials is made and certain simple precautions are rigidly maintained, there is no reason why small mines with relatively unskilled labor cannot produce a satisfactory product.

The following description outlines the technique used at Flin Flon for making carbide tipped drill steel and discusses characteristics of the brazing process that make special precautions necessary.

Drill steel is forged to four-wing shape in a conventional steel sharpening forge. Standard steel dies are modified to minimize forging cracks around the central waterhole and to forge a blunt bithead on the steel. The steel is preheated to 1500°F and held at this temperature for at least 2 min. When the temperature has equalized throughout the steel section, the drill steel is transferred to the forging furnace and heated rapidly with a reducing flame up to 2000°F. This two-stage method of heating minimizes the grain growth and decarburization of the steel while ensuring that the steel temperature does not vary greatly throughout the forging zone.

After forging the steel is allowed to cool in air to about 1600°F before being annealed in a bath of vermiculite. Despite the high hardenability of the 3 pct Ni-Cr-Mo drill steel used, this simple treatment anneals the drill steel sufficiently for milling.

The forged and annealed drill steel is slotted on a plain horizontal milling machine that is equipped with a quick opening chuck and a slot depth stop. The full depth of the slot is milled in a single pass of the 3-in. milling cutter which is fed at 3¾ in. per min across the crown of each bit wing. The slots are cut to a width of 0.342 to 0.344 in. Maintenance of this slot width is necessary to ensure that the optimum brazing clearance of 0.002 in. will result after assembling of shims and carbide in the slot.

Prior to March 1953, when the milling machine was installed, drill steel was slotted on a small manually fed ¾ hp milling attachment mounted on the bed of a lathe. Over 16,000 drill steels were slotted on this unit, and in view of its small size and low cost it gave excellent service.

Brazing of Tipped Steel

Drill steel that has been milled and cleaned in carbon tetrachloride is mounted in a rotating cradle holding six drill steels, the length of which may be from 2 to 12 ft. The slots in the drill steel, the shims, and the tungsten carbide inserts are thoroughly fluxed with a fluoride flux and assembled as shown in Fig. 1.

Fig. 2 shows the brazing equipment in use. As the ring burner is lowered over the bithead a spring valve opens the gas lines, and the gas mixture, preset to give a slightly reducing flame, is fed to the ring burner where it is lit from a pilot flame. The ring burner heats the drill steel over a zone about 1 to 2 in. below the bithead, which becomes heated by conduction through the steel.

By this means the bithead is heated rapidly and evenly, and contamination of the brazing joint with soot from the flame is avoided. The bithead is heated to the melting temperature of the brazing alloy within 1 min. This rapid heating minimizes the disadvantage of a non-eutectic brazing alloy.

The brazing alloy, a nickel-bearing quaternary alloy, is placed at the bottom of the slot below the carbide insert, as shown in Fig. 1. As the brazing alloy melts it is drawn by displacement by the carbide and by capillary action into all parts of the joint to displace liquid flux from metal surfaces.

As soon as the brazing alloy melts, each insert in turn is *wiped* by being moved back and forth along the slot. This action assists wetting of the carbide by the brazing alloy and assists in displacing molten flux from the joint.

After continuous heating for about 75 sec, when the bithead has reached a temperature of about 1500°F, the ring burner is raised and the gas supply is shut off automatically by the spring valve. As soon as heating is stopped a hand press is placed on the bithead and the inserts are squeezed down firmly. This action minimizes the clearance between the bottom of the insert and the slot. Correctly brazed steel should maintain a clearance at the bottom of the slot of 0.001 to 0.002 in.

After six steels have been brazed they are removed from the cradle and allowed to cool in air. As soon as each drill steel is cool it is dressed on a grinding wheel to remove excess flux and braze and is ground to the gage appropriate to the length of the drill steel.

T. A. O'HARA is Research and Ventilation Engineer, Hudson Bay Mining and Smelting Co., Ltd., Flin Flon, Manitoba, Canada.

Discussion on this paper, TP 3751A, may be sent (2 copies) to AIME before May 31, 1954. Manuscript, Nov. 9, 1953. New York Meeting, February 1954.

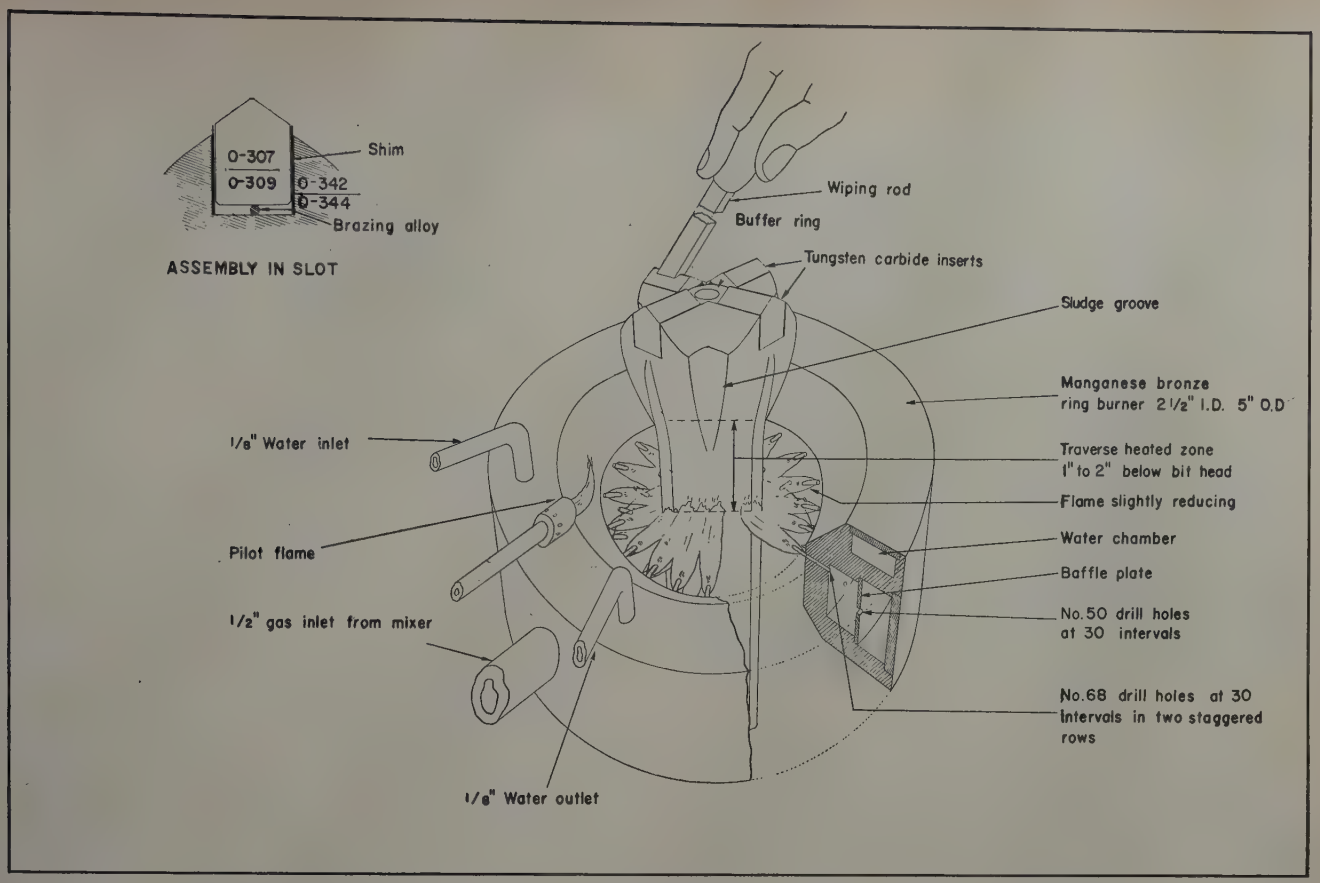


Fig. 1—The diagram above illustrates construction of the ring burner and assembly of the bithead.

The soundness of any brazed joint in a carbide bit, and consequently the performance of the bits in drilling, depends on the capacity of the brazing alloy to form a continuous solid layer between the steel and the carbide. Because of the difficulty of wetting tungsten carbide and the wide melting range of brazing alloys that wet carbide satisfactorily, a completely sound joint is rarely if ever obtained. Observations at Flin Flon on frequency of failures in commercial carbide bits and in locally tipped drill steel show that joint unsoundness is the primary cause of bit failure. Shattering or cracking of carbide tips is not necessarily caused by poor carbide, but frequently by failure of the supporting braze. Lack of soundness in a brazed joint may be brought about by any one of the following causes.

1—*Improper or Inadequate Cleaning of Carbide Inserts, Shims, or Drill Steel Slots.* All parts of the bit assembly should be thoroughly cleaned in carbon tetrachloride before assembly. At Flin Flon the brazing operator wears surgical rubber gloves when assembling inserts and shims in the bithead. This avoids contamination of the surfaces to be brazed with grease or sweat from the fingers.

2—*Improper Joint Clearance.* Long experience in industrial brazing shows that a joint clearance between 0.001 and 0.003 in. will give the soundest joint. In carbide-bit brazing, joint clearance at the sides of the slot is sometimes increased to accommodate shims for the relief of thermal stresses. Even where shims are used, the clearance between all surfaces to be brazed should not exceed 0.003 in.

3—*Improper Wetting of Carbide.* Joint unsoundness caused by improper wetting of tungsten carbide can be minimized by selection of suitable fluxes and brazing alloys and by correct technique during the brazing process.

4—*Slow Heating During Brazing.* Because all suitable brazing alloys are non-eutectic, rapid heating is necessary to minimize *liquation.* Liquation occurs when a brazing alloy separates into a low-melting fraction that is drawn into the joint, leaving behind a nickel-rich *skull* that may or may not become liquid at brazing temperature. The low melting fraction, containing little or no nickel, has poor carbide wetting ability, and joint unsoundness will therefore result.

5—*Too High a Brazing Temperature.* If quaternary alloys are heated to temperatures above 1500°F the zinc and cadmium in these alloys volatilizes, leaving gas pockets in the brazed joint, see Figs. 4a and 4b.

Thermal Stresses in the Brazed Joint

The rate of thermal expansion of tungsten carbide is about half that of steel; consequently the joint clearance of tungsten carbide inserts and the slot will increase on heating and decrease on cooling. During the austenite transformation of steel in the temperature range 1250° to 1350°F the steel shows an inversion in its thermal expansion, while the rate of thermal expansion of carbide remains constant. Fig. 3 shows the extent to which joint clearance will vary with temperature as long as carbide and steel are free to expand and contract.

It can be seen in Fig. 3 that when a tungsten carbide insert 5/16 in. wide is brazed in a steel slot at a temperature of 1200°F the amount of differential contraction on cooling will be 5/16x0.0053, or 1.006 in. A brazed layer of about 0.002 in., as required by conventional brazing practice, obviously cannot absorb more than a fraction of the differential contraction, and the remainder must be absorbed as compressive stresses in the steel and carbide adja-

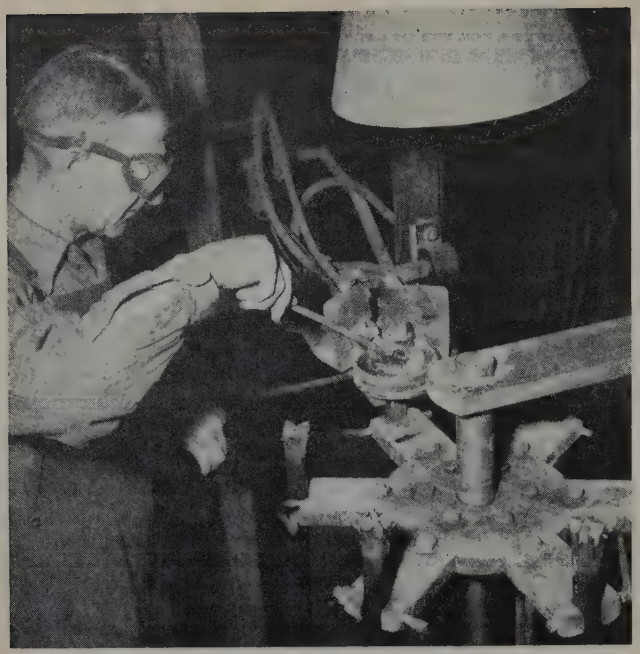


Fig. 2—Brazing the drill steel.

cent to the joint. In a joint brazed without shims, these stresses may rupture the braze, Figs. 4c and 4d.

If a brazing alloy is used that solidifies within the critical range of the steel, the total differential contraction of the joint on cooling to room temperature will be lower, about 0.0012 in. for a 5/16-in. slot. However, the rate at which the tensile stresses accumulate during the critical range is much higher than the rate of accumulation of compressive stresses during cooling below the critical range. The resultant effect will be to increase the chance of premature failure of the brazed layer.

The effect of the thermal stresses can be minimized by three methods:

1—Use of a brazing alloy which solidifies below the critical range of the steel.

2—Slow cooling the brazed assembly to minimize localization of compressive stresses next to the joint.

3—Use of a joint artificially thickened by shims of ductile material which will absorb the compressive stresses without unduly increasing joint unsoundness.

Brazing With Shims

In the *sandwich* braze, copper or mild steel shims about 0.010 in. thick are placed between two pieces of brazing alloy foil and assembled on each side of the insert in the brazing assembly. Because an insufficient amount of brazing alloy cannot be added in foil form to fill the joint completely, *topping off* the joint with added brazing alloy is necessary as the brazing temperature is reached. Use of metal shims doubles the metal surface to be cleaned, fluxed, and brazed and increases the unsoundness of the joint. Tri-Clad materials, consisting of a copper or mild steel shim clad on both sides with brazing alloy, offer advantages over the sandwich braze. Fewer pieces are required in the bit assembly, and because the shim surface is already clad with brazing alloy, joint unsoundness is reduced.

Metallurgical examinations of the brazed bond and drilling tests have been made at Flin Flon on tipped drill steel brazed with plain steel shims, perforated steel shims, iron gauze, and tri-clad shims. These tests show that the least joint unsoundness and the best drilling performance result from using tri-clad shims that have been rolled with a corrugated surface to aid capillary action.

Successful brazing of carbide tipped drill steel depends as much on wise selection of brazing materials as on correct brazing technique. Moreover, the special properties of brazing materials required for carbide-bit brazing are such that only a small group of brazing alloys and fluxes can be used successfully. The properties desired of an alloy ideally suited for brazing carbide bits are good wetting ability on steel and tungsten carbide, narrow melting range, low flow temperature, high joint strength and good ductility, and high fluidity at the brazing temperature. No one brazing alloy, of course, has all these desired properties.

The problem of selecting a suitable brazing alloy is accentuated by the use of heat-treatable steels in the bit bodies of detachable bits or in tipped drill steel. This requires that brazing be conducted at temperatures and under cooling conditions that will induce the desired hardness, strength, and toughness in the steel body of the bit. Brazing alloys that can be used successfully in carbide-bit brazing will belong to one of the following groups.

1—The Nickel-Bearing Quaternary Alloys: This group of alloys is most widely used in carbide-bit brazing and is considered to possess the best compromise of the desired properties. An alloy typical of this group is comprised of 50 pct Ag, 15½ pct Cu, 15½ pct Zn, 16 pct Cd, and 3 pct Ni, with a melting point of 1195°F and a flow point of 1270°F. This alloy has good carbide and steel wetting properties, low melting temperature, good ductility and high joint strength, and fairly good fluidity at temperatures slightly above the flow temperature. Its wide melting range is a disadvantage that must be minimized by rapid heating. At temperatures above 1500°F zinc and cadmium in the alloy volatilize to form gas pockets in the brazed joint.

2—Nickel or Manganese-Bearing Ternary Alloys: An alloy typical of this group is comprised of 40 pct Ag, 30 pct Cu, 28 pct Zn, and 2 pct Ni, with a melting point of 1240°F and a flow point of 1435°F. Alloys of this group have good wetting properties for steel and carbide, good joint strength, good ductility, and fairly easy flowing characteristics, but higher application temperature and somewhat wider melting range make them less desirable than the nickel-bearing quaternary alloys for most carbide-bit brazing applications. Their higher melting temperatures may be useful where brazing temperatures of 1500° to 1600°F are necessary to induce the required hardness and strength in the steel bit body.

3—Nickel Brasses: These alloys have a melting range of 1700° to 1850°F. Analysis of a typical alloy is Cu 46½ pct, Zn 42½ pct, Ni 11 pct. These alloys have a strong wetting action on steel and carbide and form very strong joints, but these characteristics are offset by the high thermal stresses induced in the joint by cooling from the high brazing temperatures required. The sluggishness of these alloys throughout their melting range and the lack of ductility in the brazed joints formed restrict their usefulness for brazing carbide bits.

4—Copper: Copper has been used for brazing carbide tips in furnaces with reducing atmospheres, but its high melting temperature (1982°F) makes it unsuitable for torch or induction brazing.

Carbide tips are almost always brazed in air; consequently fluxes must be used to remove the oxides formed on the steel surface and to assist the wetting

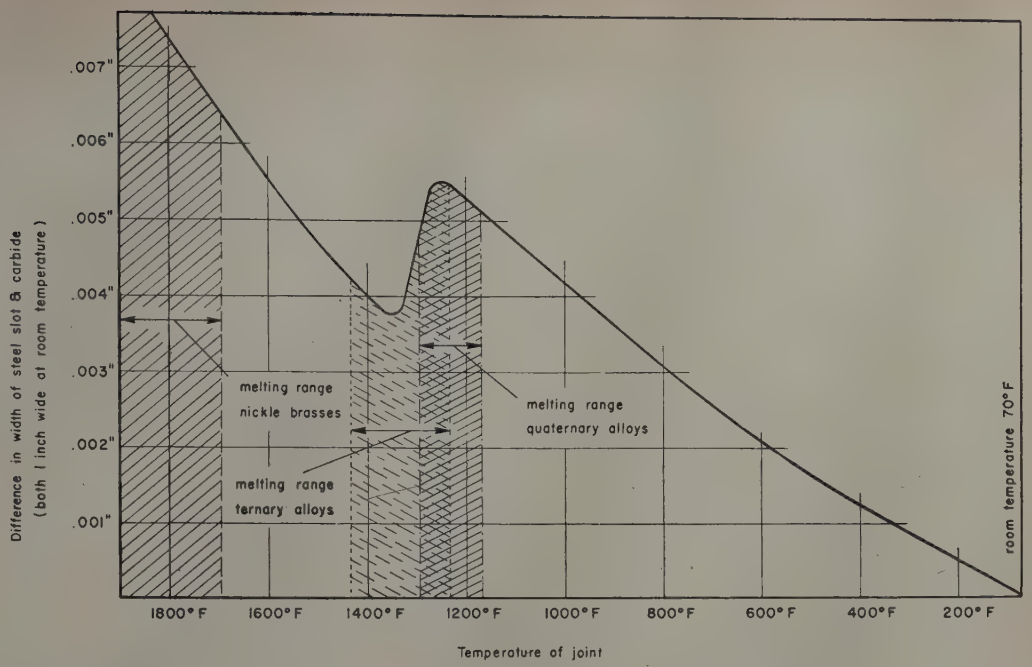


Fig. 3—The graph at left illustrates the differential contraction of steel and carbide.

of the metal surfaces by the brazing alloy. Although a flux with a low melting temperature is desirable to minimize oxidation of the metal surface, such low temperature fluxes are unstable at higher temperatures. In practice the most suitable fluxes melt at temperatures about 100°F below the melting point of the brazing alloy.

Fluxes

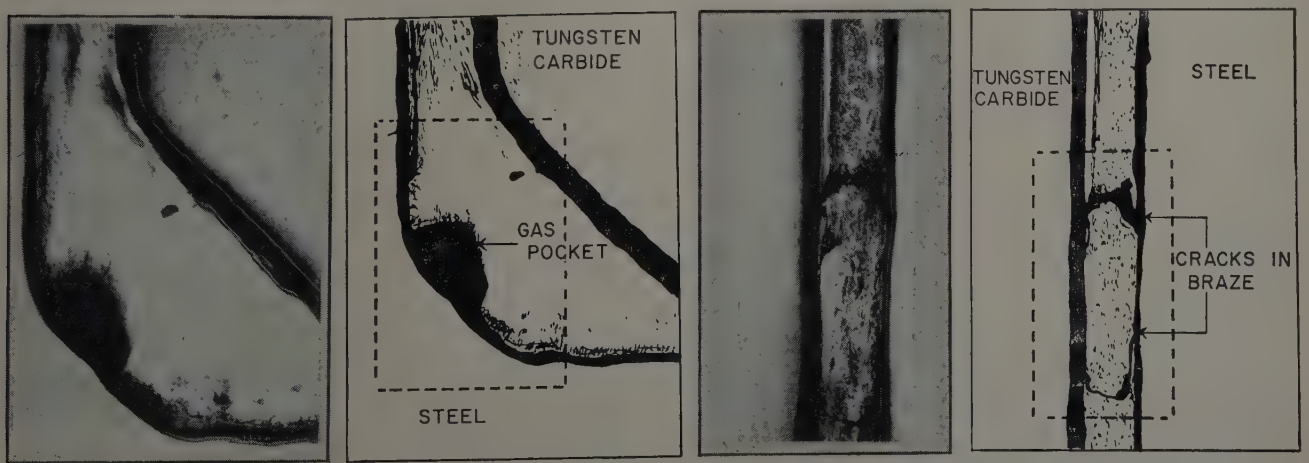
Fluoride fluxes are ideally suited for brazing with quaternary and ternary brazing alloys at temperatures less than 1500°F. At higher temperatures these fluxes are not stable and evolution of fluoride vapor causes undesirable working conditions. They are much stronger oxide solvents than the borax base fluxes. Fluoride fluxes containing sodium have an undesirable yellow glare when heated and fluxes containing potassium instead of sodium are to be preferred.

Borax-boric acid fluxes are very fluid when molten but are not strong oxide solvents, and they become sluggish when laden with oxides. These fluxes are useful in brazing with alloys which melt above 1500°F, but at lower temperatures the fluoride fluxes are much more suitable.

Tungsten carbide tipped drill steel must be cooled slowly from the brazing temperature if cracking of the carbide insert or braze failure is to be prevented. If cooling is too slow, however, the drill steel will not harden sufficiently to provide rigid support under the inserts. Tests at Flin Flon on drill steel cooled at different rates, resulting in Rockwell C hardness of from 24 to 53, show that satisfactory performance in drilling can be expected only when the hardness of the drill steel around the inserts is at least Rockwell C 40.

To obtain the high hardness required in the steel at a slow cooling rate, it is necessary to use an air-hardening drill steel. The drill steel used at Flin Flon is a medium carbon nickel alloy steel having a composition of 0.43 pct C, 0.60 pct Mn, 3.00 pct Ni, 0.40 pct Cr, and 0.25 pct Mo. This drill steel hardens satisfactorily by air cooling from 1500° to 1550°F to a Rockwell C hardness of about 50. The type of alloy steel formerly used at Flin Flon in shanks for detachable steel bits was a high carbon chrome alloy steel with the following composition: 0.95 pct C, 0.30 pct Mn, 1.00 pct Cr, and 0.25 pct Mo. Although the as-rolled hardness of this grade of steel is somewhat higher than the medium carbon nickel drill steel it does not air-harden as well.

Although the high carbon chrome type of drill steel is used extensively by Swedish tipped drill



Figs. 4a and 4b (left) illustrate a large gas pocket at corner of steel slot. Original magnification of Fig. 4a X100. Reduced approximately one-half. Not etched. Figs. 4c and 4d (right) illustrate rupture of braze resulting from thermal stresses in a bit brazed without shims. Original magnification of Fig. 4c X100. Reduced approximately one-half. Not etched.

steel manufacturers, it is believed that the medium carbon nickel drill steel is more suitable for torch brazing with quaternary silver brazing alloys.

It is well known that steel must be heated above the critical temperature and *soaked* at that temperature before it will harden completely on cooling. Soaking of the steel above the critical temperature is necessary because the steel must be completely transformed to austenite before it can be hardened on cooling. Alloying elements that form carbides, such as chromium and molybdenum, retard the austenite transformation. Thus alloy drill steels that derive their air-hardening power from alloyed chromium and molybdenum must be soaked for several minutes or heated to temperatures above 1600°F before they will air-harden satisfactorily.

On the other hand, alloy drill steels that air-harden chiefly because of their nickel content can be transformed to austenite at lower temperatures (1500°F).

Before tipped drill steel is placed in service at Flin Flon it is dressed, gaged, and hardened at the chuck end. Brazed drill steel is dressed on a C60 M5 VBE grinding wheel to remove excess braze, flux, and forging irregularities. Each insert is ground lightly on its outer edge to blunt the outer cutting edge. The outer periphery of the bithead is ground at 3° gage angle to the required gage on a 37 C60 K8 VK silicon-carbide grinding wheel. The required gage varies by 0.030 in. for each standard length of drill steel, being larger for the shorter lengths of drill steel. This ensures that new drill steel will not bind in a hole which has been drilled by a shorter worn drill steel. The standard gages for new drill steel are: 2 ft 6 in. steel, 1.50 in. gage; 4 ft 6 in. steel, 1.47 gage; 6 ft 6 in. steel, 1.44 in. gage; 8 ft 6 in. steel, 1.41 in. gage.

The chuck end of the drill steel is hardened to about Rockwell C 50 hardness by heating to 1475°F in pyrometer-controlled tube furnaces and cooling in air. The tipped drill steel is then placed in service. Tipped drill steel dulled in drilling is resharpened on a 37 C 60 K8 VK silicon-carbide grinding wheel.

Drill steel failures in service may be due to wearing out of carbide, loss of carbide, shattering of carbide, failure of steel around carbide, or breakage of the steel shank at the neck of the upset portion. The damaged end of these drill steels is cut off, serviceable inserts are salvaged, and the steel shank is reforged and re-tipped. Drill steel that has broken in the center of the shank away from the heat-affected zones on the bit and chuck ends and steel that has been shortened to 2 ft 0 in. long because of successive re-tipping is discarded. Eighty-eight per cent of all tipped drill steel made is used drill steel that may be re-tipped several times before being discarded.

The usual length of hole drilled at Flin Flon is 7 ft. Thus drifters with a 4-ft steel change can accommodate starters 3 ft 6 in. to 4 ft 6 in. long and followers 7 ft 6 in. to 8 ft 6 in. long. Stopers with 2-ft steel change require changes of 2 ft or less, and usable lengths of steel will include the following sizes: 2 ft 0 in. to 2 ft 6 in., 3 ft 9 in. to 4 ft 6 in., 5 ft 6 in. to 6 ft 6 in., and 7 ft 6 in. to 8 ft 6 in. Drill steel that is 1 ft shorter than the standard lengths of 2½, 4½, 6½, and 8½ ft is seldom useful in drilling, and this intermediate length will accumulate on the steel racks. Periodically this steel is cut down to the nearest standard size. Thus a new drill steel 8 ft 6 in. long will, on successive retippings, have lengths of 8 ft 3 in., 8 ft 0 in., 7 ft 9 in., and 6 ft 6 in., 6 ft 3 in.

Many mine operators believe that a drill steel becomes *fatigued* and useless for further drilling as soon as the inserts are worn out. Although there is very little support for this in practice or in theory, it serves to relieve the misgivings of mine operators when they accumulate a stock of used tipped drill steel. Some mine operators grind the bit end of this used steel to accommodate steel detachable bits, but the advantages of carbide drilling are lost. Retipping this used drill steel in the mine shops would be a practical way to utilize it. Most of it, unfortunately, is a high carbon chrome alloy drill steel, which is not as suitable as medium carbon nickel drill steel for tipping with a torch or ring burner.

All drill steel required for the Flin Flon and subsidiary mines is tipped in the Flin Flon drill steel shop. The average number of drill steel tipped monthly by a crew of five is 2037. The cost per tipped drill steel during 1952 was $6.40, which includes the cost of four carbide inserts. The average cost of four new inserts is $3.72, but carbide cost per drill steel is lowered somewhat by re-using salvaged inserts that are in good condition.

Drilling costs, including cost of tipping drill steel, sharpening, and nipping, averaged 7.7¢ per ft drilled during the first eight months of 1953. Table I shows the drilling costs and performance for the last six years. This table illustrates the increase in drilling performance since 1948, resulting chiefly from increased use of tungsten carbide bits or local tipped drill steel. Drilling costs increased when purchased carbide bits were used, but since 1952, when the local tipped drill steel was used for nearly all drilling, drilling costs have declined sharply.

Table I. Costs and Performance in Drilling at Flin Flon Mine

Year	Drilling with Tungsten Carbide, Pct	Footage Drilled per Driller Shift	Cost per Foot Drilled, ¢
1948	0.8	49.9	9.2
1949	18.5	51.5	9.9
1950	54.6 (Incl. 0.9 pct local)	61.5	11.6
1951	50.6 (Incl. 17.4 pct local)	69.2	11.0
1952	90.5 (Incl. 89.2 pct local	74.2	7.0
1953 (8 months)	99.8 (All local tipped)	83.5	7.7

Equipment necessary for carbide tipping drill steel need not be costly. Most mines have a forging furnace and a steel-sharpening forge, which can be utilized in forging the bithead. Few small mines have a milling machine, but unless a large amount of carbide tipped drill steel is required a small milling attachment should be adequate. Several small manually fed millers or milling attachments are available at a cost of $350 to $1200.

The conventional acetylene torch may be used for brazing carbide tips, although a ring burner will be much more satisfactory. The water-cooled ring burner shown in Fig. 1 was made in the mine machine shop and it is understood that somewhat similar burners are now available from suppliers of welding equipment. Induction heating is cleaner and faster than acetylene heating, but unless a large output of brazed drill steel is required the high cost of suitable motor generator equipment is not justified. Joints brazed carefully with a suitable gas burner need not be inferior to those brazed with induction equipment.

Trends in Coal Utilization and Their Effect On Coal Marketing

by Carroll F. Hardy

The day by day loss of industrial plants to gas and oil is chiefly by default. The coal industry is not selling its superior economy, safety, and other advantages to its customers.

THE position of the coal industry has been affected by a wide variety of developments in the production and use of energy. The tempo of development and change has been increasing and the end is not in sight. Legislation is currently being proposed for commercial use of atomic power, and the employment of atomic energy in significant quantity will probably occur about the same time as the decline in production of petroleum and natural gas. But these developments are in the future and have little immediate effect on utilization and marketing of coal. While no one should try to suppress or retard the development of a new and economical source of energy, both the coal and private utility industry should be allowed to question how the nuclear power is to be used, who is to use it, and who is going to pay for it. The taxpayers have a monopoly on fissionable material and the knowledge to employ it. Any commercial use must stem from this source. It is not hard to visualize either taxpayer-subsidized private utility atomic power plants on one hand and super TVA's on the other.

In view of the gains of gas and oil in the home heating field, it is interesting to compare the 1940 and 1950 census reports on the kind of fuel used for heating in occupied dwelling units. Table I shows that whereas coal provided 77 pct of the fuel for central heating (furnaces and boilers) in 1940, it was down to 45.4 pct in 1950. However, only about 1½ million units were lost in this 10-year period.

In the non-central heating category, which principally includes stoves, the percentage declined from 39.2 to 25.6, but the units declined about 2½ million in number. The big increase was in heating units designed to burn gas and oil. Use of wood for central heating declined about one-third.

Data on amount of fuels used for residential heating are not available, but information is on hand for residential and commercial space heating, see Table II. Commercial space heating includes office buildings, churches, schools, and similar structures. The annual use of bituminous coal in these two categories declined about 1 million tons in the 10-year period. Other forms of solid fuel showed greater losses, except wood, which remained the same.

C. F. HARDY, Member AIME, is Chief Engineer, Appalachian Coals, Inc., Cincinnati.

Discussion on this paper, TP 3752F, may be sent (2 copies) to AIME before May 31, 1954. Manuscript Nov. 2, 1953. Meeting of Central Appalachian Section AIME, Somerset, Ky., June 1953.

Domestic stokers reached their high point in 1948 with about 1,200,000 in use. At the end of 1951 there were approximately 1,116,790 stokers in use. Conversions to gas and oil have been from hand-fired heating plants in the ratio of about 7 to 1 compared to stokers. In other words, for every one stoker which has been converted to gas or oil, seven hand-fired units have been converted to gas or oil. A bare recital of these data would indicate that the coal industry is holding its own reasonably well. However, 93.4 pct of the new homes built in 1951 were heated by gas or oil. Oil-burning equipment was installed in 37.8 pct and gas equipment in 55.6 pct of the new homes. This indicates that the public prefers gas when it is available, and that oil is second choice, with all forms of solid fuel apparently used when it is unavoidable.

It must be pointed out, however, that during the period of rapid expansion of gas pipelines gas has been sold for house heating at prices that are in some cases actually lower than coal prices, or very nearly on a par. Gas has been sold at wells at far below the comparable price for oil produced from the same wells, and far below its actual worth. This situation is being remedied at the present time by increases in gas prices at the wells. For example, the wellhead price of gas in Texas averaged 7.49¢ per Mcf in 1952. In 1949 it was 4.59¢ per Mcf. This increase in price is being reflected in pipeline gas prices, and in most of the markets served by the pipelines the tendency is to get it out of the *bargain basement* type of sales.

The American Gas Association estimates that at the end of 1952 there were in the United States about 11 million customers for gas house-heating, and the Association expects additional gains each year until around 18 million homes will be heated by gas in 1975. By 1975 there should be 60 million dwelling units to be heated in the United States, if dwelling units increase at the same rate as the population. If the gas industry heats 18 million dwelling units by that time, this still leaves 42 million units to be heated by some other fuel. If oil is used to heat 18 million dwelling units in that same year, 24 million would of necessity be heated by coal, coke, wood, electricity, or another fuel. The total number of dwelling units using coal listed in the 1950 Census was 18,776,000, so it would appear that coal has a chance at least to stand still in the tonnage sold for domestic use.

In the first quarter of 1953, 2044 domestic stokers

were sold. This equals 25 pct of the sales in 1949. To recover the former sales volume the coal industry must actively promote and sell the present units and also develop and promote units more acceptable to the public.

Trends in Industrial and Commercial Utilization

There are two principal trends in the general industrial and commercial fields. One of these is the trend away from hand-firing and the handling of coal by shovel and wheelbarrow, and the other is away from coal. Only a few industrial plants are still hand-firing coal, and these in general are in seasonal industries such as canning factories, or smaller and older plants. It was long felt that in as much as a fireman had to be on duty in any case, he might as well hand-fire the coal and move coal and ashes by wheelbarrow. However, because of the stoker's superior efficiency, this is not a valid assumption. It is also true that younger men, particularly, do not care to work in a hand-fired plant. They prefer a less arduous job.

Table I. Heating Fuel for Occupied Dwelling Units in the United States Urban and Rural for Years 1950 and 1940

Central Heating	1950	Pct	1940	Pct
Number reporting fuel	20,724,000	100	14,152,024	100
Coal	9,430,000	45.5	10,903,163	77
Wood	250,000	1.2	373,322	2.6
Gas	5,914,000	28.5	1,109,587	7.8
Utility gas	5,719,000	27.6		
Bottle gas	195,000	0.9		
Liquid and other fuels	5,130,000	24.8	1,765,952	12.5
Liquid fuel	4,678,000	22.6		
Other fuel	452,000	2.2		
Non-Central Heating	**1950**	**Pct**	**1940**	**Pct**
Number reporting fuel	20,053,000	100	19,469,707	100
Coal	5,127,000	25.6	7,622,427	39.2
Wood	3,969,000	19.8	7,362,155	37.8
Gas	5,881,000	29.3	2,728,381	14.0
Utility gas	5,204,000	26.0		
Bottle gas	676,000	3.4		
Liquid and other fuels	5,077,000	25.3	1,756,740	9.0
Liquid fuel	4,437,000	22.1		
Electricity	272,000	1.4		
Other fuel	369,000	1.8		

The same general thought applies to stoker-fired plants where coal must be wheeled to the boiler room and then shoveled into the stoker hopper. Coal-handling equipment has proved its value, even in those plants where there is a minimum labor force. The fireman has a chance to concentrate on keeping the stoker running at top efficiency, and should, in theory at least, cut down on the waste of fuel by inefficient operation.

In the smaller plants, automatic ash-handling equipment cannot be justified because of the relatively few ashes to be handled. In the medium-sized and larger plants, some types of ash-handling equipment usually can be justified.

The trend away from coal towards gas and oil is caused by the same factors behind the trend toward gas and oil in domestic heating. Increases in mine and freight costs, strikes, labor costs, smoke abatement activity, trouble in the use of coal due to improper selection, poor coal deliveries—all are reasons for dissatisfaction. A dissatisfied user is a poor prospect. In many plants management has been plagued by undersized boilers, improper design, worn-out equipment, and a whole parade of incompetent firemen, as well as by direct troubles with coal. A *packaged boiler* which has complete control and may be started and stopped by push button offers quite a contrast. The boiler is delivered to the plant with the burner and controls already in it. It is skidded into place, the oil or gas line and the steam line are attached, the stack connection is made, and it is ready to go. Management is often told that no supervision is necessary, that there will be little or no maintenance, and that once such an installation is made their problems are all solved. They are assured that aside from an occasional check as an extra precaution, the boiler room will be self-operating.

The packaged boiler has had a very wide acceptance, in fact, a much wider acceptance than an examination of all the factors involved would warrant. It is not maintenance-free, nor is it foolproof. It is true that the packaged boiler designed for gas or oil is lower in first cost than a standard boiler and stoker combination complete with automatic controls and automatic coal-handling equipment. However, it is often overlooked that a saving of 12 pct, for example, in investment cost will be wiped out in a year or less by the higher cost of the fuels used. In most of the industrialized part of the United States, coal is by far the lowest cost fuel.

In those areas where gas is available on an interruptible basis, it can sometimes be shown to be economical to burn. However, there must be a standby fuel in this case. Oil is used for standby in packaged boilers, on the assumption that the gas will be off for only a short period of each year and that the high cost of oil, because of the small quantity used, will be of no consequence.

The price of interruptible gas is usually tied to coal, and gas is sold at about the same price or perhaps a little less than the cost of coal. Oil, except in a narrow area on the eastern seaboard, is usually one and a half to two times as expensive as coal. Therefore, when gas is not available and oil must be used, apparent savings of using gas are quickly overcome. In cases where gas is competitive, coal can be used as a standby at somewhat higher cost installation than a straight packaged boiler, and full economy is effected. It has been true for many years that the amount of gas available on an interruptible basis dwindles as the pipeline is loaded up. To design a boiler which may be in use for 30 years or more to use interruptible gas with oil standby is sheer folly, whether it is a packaged boiler or a standard boiler.

There are many cases on record where packaged boilers have been put in and then taken out when they proved uneconomical, with a loss to the customer which could have been prevented had the full facts been known. Standard boilers installed to use gas or oil and not designed for easy conversion to coal present a serious problem also. It is possible to use coal in a modern fully equipped installation with no more labor force than is required in a combination boiler burning gas and oil. Until this fact is firmly established in the minds of consulting engineers, architects, and fuel users, the industry will continue to lose business in this group. The whole coal industry must expend more effort in *education* and promotion to stop or even slow down the trend away from coal in the industrial field.

The effect of the partial dieselization of all but one of the country's major railroads and of most of the smaller railroads is well known. It has eliminated much of the run-of-mine, resultant, and double-screened tonnage that was sold for locomotive fuel. The trend towards eliminating the coal-

fired steam locomotive has certainly not stopped and may not stop until some of the major railroads are completely dieselized.

In addition to providing a market for about one-fifth the total bituminous coal produced, consumption of railroad fuel had another important function. The practice of allowing the producer to ship almost any size of coal for locomotive fuel was an accommodation to the producer and allowed him to get rid of no-bills and canceled cars. This balance wheel, generally speaking, has been lost, in addition to the loss of 120 million tons of railroad fuel.

Table II. Fuel Consumption for All Residential Uses and for Commercial Space Heating, According to Physical State at Point of Combustion for Years 1940 and 1950

Liquid Fuels (Thousands of Barrels)	1940	Pct	1950	Pct
Distillate grades 1, 2, 3, and 4	115,533	10.1	220,947	14.7
Range oil No. 1, fuel oil, and kerosene	44,692	3.8	94,662	6.2
Residual grades 5 and 6	44,846	4.3	72,716	5.3
Total	205,071	18.2	388,325	26.2
Gaseous fuels (millions of therms)				
Natural gas				
Residential	4,064.3	6.1	11,561.6	13.2
Commercial	1,226.2	2.7	3,496.6	4.0
Manufacturing gas including mixed gas				
Residential	1,758.8	2.7	2,277.5	2.6
Commercial	371.5	0.6	607.2	0.7
Liquefied petroleum gas	128.0	0.2	1,931.5	2.2
Total	7,548.8	11.4	19,874.4	22.7
Solid fuels (wood, thousands of cords; all others thousands of net tons)				
Bituminous and lignite	87,700	34.6	86,604	26.0
Anthracite	39,300	15.1	29,558	8.6
Coke	8,231	3.2	2,560	0.8
Briquets and packaged fuels	1,313	0.5	2,730	0.7
Petroleum coke	844	0.4	1,201	0.4
Wood, including mill waste	48,500	15.3	48,739	11.7
Total		69.1		48.2
Electricity (thousands of kilowatt hours)				
Produced from fuel	16,865,569	0.9	52,733,944	2.1
Hydro	8,442,908	0.4	21,696,464	0.8
Total	25,308,477	1.3	74,430,408	2.9

The rapid expansion of electric utilities is an old story although an almost unbelievable one. The expansion of electric utilities in the eastern half of the United States will require between 30 and 40 million tons of additional coal by 1956, if all the units planned are built. This is a very encouraging trend, but not a completely happy condition.

Over a 25-year period the electric utilities have reduced rates very materially and have maintained these low rates even in the face of rising costs of labor, fuel, and other materials. Part of this has been done by the improvement in fuel-burning equipment. This is shown by the amount of coal required to produce 1 kw-hr of electricity. In 1930, for example, it took 1.6 lb of coal per kw-hr; in 1940, 1.34 lb per kw-hr; in 1950, 1.19 lb per kw-hr; in 1952, 1.10 lb per kw-hr, and in March 1953, 1.08 lb per kw-hr. The effect of this reduction in coal rate from the 1.19 lb average in 1950 to the 1.08 lb average in March 1953 is just over 10 pct. That is, if fuel required per kilowatt-hour in March 1953 had been at the 1950 average rate, 1,600,000 more tons (or equivalent) would have been used. On the basis of a full year at this rate, over 19 million tons more coal or coal equivalent in gas and oil would be required. This reduction in fuel requirements per kilowatt-hour has been common to all three fuels and has been accomplished by higher steam pressures and temperatures, better cooling of electric generators, better combustion controls.

The most modern steam electric plant has a heat conversion efficiency of 37 pct. In other words, it uses 37 pct of the heat in the fuel. Announcement was recently made by the American Gas and Electric Co. of a boiler designed to operate at 4500 psi steam pressure and 1150°F. This boiler will be much more efficient than the present boilers, although the exact efficiency is not revealed.

The trend in designing furnaces has been towards more liberal or flexible design, so that a wide variety of coals may be burned, more specifically, so that the lower-grade coals may be burned and the pressures still be maintained on the boiler. This trend, which started in the public utilities, has gradually spread to large and small industrial plants. Thus as the coal industry has spent $2 billion in the past 10 years in underground and surface production facilities and cleaning plants, to give a better and more uniform product, the coal customers have spent an equal or greater amount for equipment which would use a lower-grade fuel. Part of this trend for wider flexibility has stemmed from the desire to use locally produced coals and thus save the freight rate on better-grade coals brought in from a distance.

Large industrial plants have followed the lead of the utilities in boiler design and coal-buying practices. Both classes of plants have had troubles such as slagging of tube banks, clinkering, and coal and ash handling difficulties, from using poor coal. Large utility plants have been put out of service by low-grade coals. In less extreme cases extra maintenance and lower operating capacity have cost more than the savings on the delivered cost of the low-grade coal versus good coal.

The Trend of the Delivered Price of Coal

Table III is a compilation of the coals bought by a large midwestern plant using approximately 280,000 tons of coal per year. The plant was new and modern in 1937 and the equipment is essentially the same today. The same type of coal has always been used in this plant, as coal selection is somewhat limited by furnace design.

There are several interesting points about this table. Column *A* gives the yearly average Btu per pound as bought. In 1937 the average was 13,575; in 1938 it had risen to 13,790. The low point was in 1949 when the average had dropped to 12,865. In 1952 it was 13,175. There have been many rumors about the decline and quality of better-grade coals because good seams are all mined out or because mechanical mining has supplanted hand loading. Actually, those who spread the rumors know that this is not the case. High-quality coals are as available today as they were before World War II. There have been some changes in quality of individual mines, and some mines in good seams have mined out as have mines in poor seams. In the case of a particular plant, after the demise of fixed prices and controls on coal, higher Btu coals than the ones used were available. However, it was the decision of management to use the somewhat lower cost and lower Btu coals.

Column *B* of Table III shows the average mine price paid during the 16-year period. It will be noted that the mine price has declined exactly $1.00 per ton between 1948 and 1952.

Column *D* shows that the average delivered price has declined only 32¢ in that same period, although owing to increases in freight rates the delivered price increased in 1949 over 1948.

Column C shows an increase in freight over the 15-year period since 1937 of $1.55 a ton. The present freight rate is $3.66, so by the end of 1952 the freight had increased 75¢ a ton over the rate in 1948. In this same four-year period, the cost of labor at the mines had increased $1.05 a ton. The average delivered cost per ton in 1952 was 32¢ less than it was in 1948 and 48¢ less than it was in 1949. The producers have received nothing for their increase in labor cost since 1948 and have in effect absorbed the increase in freight so that they are receiving the equivalent of $2.12 less per ton than they were in 1948. The profit or margin in 1948 was only a small part of this $2.12, so that the producer has had to cut production cost in many ways even to stay in business.

As C. J. Potter recently pointed out, with the tonnage off about 28 pct from the peak year of 1947, and with prospects of further decreases, many coal operations have had to go out of business. Stronger elements, as mentioned above, have made major investments in mining and preparation facilities and major reductions in labor costs. The financially weaker elements of the industry have turned to opportunists' methods of mining, or have attempted to operate non-union, or have closed.

With production off 70 million tons since 1951 and prospects for further decline in tonnage for 1953, together with the fact that the coal industry's profits are approaching or below the zero point, a revolution of coal mining or marketing must, of necessity, be in the making.

The Effect on Coal Marketing

A purchasing agent said recently, "Coal marketing consists of trying to cut the price and go broke quicker than the other coal companies can do it."

Table III. Cost and Heating Value of Coal Used by a Large Midwestern Plant, 1937 to 1952 Inclusive

Year	Avg Btu Per Lb	Avg Mine Price, $	Avg* Freight, $	Avg Deliv. Cost Per Ton, $	Avg Cost Per MM Btu, ¢
	A	B	C	D	E
1937	13575	1.69	2.04	3.73	13.75
1938	13790	1.64	2.14	3.78	13.70
1939	13650	1.59	2.14	3.73	13.65
1940	13750	1.61	2.14	3.85	14.00
1941	13635	2.17	2.14	4.31	15.75
1942	13615	2.34	2.23	4.57	16.80
1943	13470	2.70	2.23	4.93	18.30
1944	13275	3.01	2.23	5.24	19.75
1945	13225	3.04	2.23	5.29	20.00
1946	13185	3.30	2.37	5.67	21.50
1947	12985	4.19	2.58	6.82	26.25
1948	12915	5.23	2.91	8.14	31.50
1949	12865	5.08	3.22	8.30	32.25
1950	13200	4.56	3.27	7.83	29.65
1951	13165	4.56	3.36	7.92	30.08
1952	13175	4.23	3.59*	7.82	29.89

* All increases in freight rates were applied as they became effective and the costs averaged for the year. The 4¢ transportation tax is included.

Coal salesmen are continually offering to cut prices to move coal, or are ready to meet any cost per million Btu figure regardless of the actual use value of the coal. Purchasing agents are naturally either skeptical about the fact that the coal industry is not making money or think they might as well get in on a good thing as long as coal is being sold at a loss. The fact that coal cannot continue to be sold at less than the cost of production does not worry them, since they are regularly offered more tonnage than they can use, at lower and lower prices. This inept marketing on the part of the coal salesmen has encouraged many buyers arbitrarily to set a cost per million Btu and hold all suppliers to this price regardless of coal quality. They are trying to beat down the prices which are already too low. These customers of the coal industry evidently do not realize that much of the reduced cost of mining coal in the last few years has seriously harmed mine operations. It has produced no capital for replacement or renewal and has paid nothing for the money used. The present over-capacity of the coal industry has permitted this disorderly marketing —if it may be called marketing—to exist, and has permitted the buyer to write his own ticket.

Fortunately, not all purchasing agents and their managements feel this way about coal. They have given some consideration to the future supply of coal as well as to the present situation. In a recent comprehensive statement on this subject, Robert E. Dennis, Fuel Agent of the Consolidated Edison Co., said in part:

> So let us recognize that coal is our basic, most dependable source of energy for static power plants. And let us see to it that the coal industry is not so injured by loss of business to liquid and gas fuels as to make it impossible for it to absorb a large and rapid demand to supply the energy needs of our basic industries, which employ static power plants.
>
> As the needs for more power come upon us in the future, it's a cinch that coal will have to supply the bulk of it. What we have to do now and perhaps next year, is to help the coal industry over this period. After that, it is my opinion coal *can* be able to meet all competition, due to the demand for coal from the utility and chemical industries. To keep the coal industry healthy and competitive, it needs, above all else, tonnage sufficient to meet its inherent mining load factory. . . . In the first and most important place, there is need for recognition on the part of all industry that, in harming coal they are jeopardizing the future security of this country, and of course, themselves. Of what use are millions upon millions of tons of coal near at hand, with no one willing to put up the money, no one willing to put in the labor to obtain it? I am the buyer for a company whose needs for fuel are enormous, and whose struggles for more economical ways to give better service are unceasing. Nevertheless, we see clearly that today's economy may be tomorrow's disaster and, we must, therefore, have strong, efficient industries to supply and transport our fuels and, in so far as we can do so, help them to stay that way.

The coal industry is faced with the task of educating all consumers of coal towards full realization of the true conditions of the industry and the consequences of irresponsible buying and selling of coal.

The Task of Marketing Coal Properly

The consumer of coal must be brought to realize that a large proportion of the producing capacity of the industry is made up of thoroughly reliable companies which can be depended upon in any emergency. This might be three-fourths, or more, or less, of the industry. These are the companies that have an investment in the industry in the form of adequate reserves, adequate mining, and preparation machinery. These are the people who make every effort to mine coal safely, prepare it properly, and market it consistently.

The rest of the industry is made up of opportunists who saw a chance to get into coal production during the war and when new tonnages opened up, for example, the T. V. A. tonnage. Their only interest is to try to make a quick dollar by excavating and selling coal without preparing or standardizing.

In this day of exact purchasing, of careful testing, of micrometer measuring, the method of coal buying in most companies is badly out of step with the rest of the material procurement. The steel used by a company is usually bought on specification from a large steel company. It is then tested and analyzed to make sure that it is up to specification before it is put in the product. Every effort is made to purchase from reliable, established companies. The jobber with a few tons of steel or a few barrels of unbranded paint has little chance of selling to a careful buyer. The buyer would certainly want the analysis, the origin of the material, and probably the reason it was on the market at reduced prices.

Table IV. Comparative Annual Fuel Costs in a Plant Requiring 695.5 Billion Btu Annually

Coal Analysis, As-Received	COAL A 1¼x0 In.		COAL B 1¼x0 In.
Moisture, pct	3.95		6.18
Ash, pct	3.97		9.55
Sulphur, pct	0.70		4.49
Ash softening temp., °F	2700		2070
Btu per lb	13910		11740
Operating Costs			
Tons of coal required	25,000		29,620
Mine price of coal, $	4.70		2.90
Freight rate, $	4.70		4.70
Deliv. cost per ton, $	9.40		7.60
Deliv. cost per million btu, $	0.3379		0.3237
Total deliv. fuel cost, $	235,000.00		225,112.00
Storage loss, weathering, fires, etc. (3 M tons stg.) 5 pct, $	141.00	15¢ per ton	3,420.00
Unloading and handling labor, 30¢ per ton, $	7,500.00	30¢ per ton	8,886.00
Coal handling equip. maintenance, 5¢ per ton, $	1,250.00	15¢ per ton	4,443.00
Soot blowing and slag removal, 1¢ per ton, $	250.00	5¢ per ton	1,481.00
Pressure parts and fly-ash collection erosion, 2¢ per ton, $	500.00	8¢ per ton	2,369.60
Corrosion of fly-ash collector, fans and stack, $		Per year	2,000.00
Ash-handling equip. operation and maintenance, 1¢ per ton, $	250.00	5¢ per ton	1,481.00
Fly-ash return system maintenance, 1¢ per ton, $	250.00	6¢ per ton	1,777.20
Grate and feeder maintenance, 3¢ per ton, $	750.00	14¢ per ton	4,146.80
Total operating cost, $	10,891.00		30,004.60
Total fuel and operating cost, $	245,891.00		255,116.60
Utilization cost per ton, $	9.84		8.61
Utilization cost per million btu, ¢	0.3537		0.3670
Annual Savings by Using Coal A, $	9,225.60		

Coal buying, unfortunately, is a different proposition with many of these same buyers. They appear to care little about the reliability of the company or even the source and analysis of the coal, and they evince small concern as to the probable result of using coal in the plant, or the extra cost that will ensue when a number of different coals are mixed together. It is not at all unusual to find a plant using coals varying in ash content from 3 to 12 pct, in moisture from 4 to 15 pct, and in Btu content from more than 14,000 per lb to 11,000 per lb in a random mixture as the coals are received at the plant. Even the best of operating personnel with the best equipment is sorely taxed to operate a plant properly under these conditions. There is a trend towards avoiding any long-term relationship with well-established coal companies, but dependence on day-to-day purchases from anyone with a few cars of coal to sell.

A recent study of a moderate-sized plant with flexible coal-burning equipment is shown in Table IV. This illustrates the difference between the cost of coal f. o. b. the plant and the complete cost of using that coal removing the ashes. The costs given in this table are based on actual experience and all estimates shown are conservative. Even in other cases where the low-ash, low-sulphur, high-Btu coal might be shown to be slightly more expensive, the human relations or good labor policy should be well worth this small cost.

Formerly it was the practice to build a factory for only one purpose—to get out production. No attention was given to the architecture of the plant or the appearance of its surroundings. If it happened to be an eyesore, as most factories were in those days, this was merely considered part of the price of having it in that location. Today this idea has given way to the esthetic or *good neighbor* policy. Factories are carefully designed and grounds landscaped. Usually both improve the neighborhood.

The purchase of good standard coal from reliable coal companies is only another step in this program. A large part of the air pollution can be prevented in this manner, and certainly the advantages well offset any slight addition in cost.

Summary

The trend in coal utilization is definitely towards use of the smaller sizes of coal. It is probable that as time goes on this will be even more pronounced. To stay healthy, the coal industry must realize enough money from the sale of these sizes to make a profit. Profit is the only known reason for private capital being invested in any operation. The coal industry's customers must be made to realize that a healthy coal industry is not only an asset but a necessity to all American industry. The alternative is wholesale liquidation of the coal industry which would be disastrous to the economy of the country.

The coal industry cannot hope to cut costs by mechanization, selective mining, augers, continuous miners, or stripping as fast as the realization can be cut by poor marketing and incorrect application.

Losses of residential and commercial heating tonnage to gas and oil are serious, although new equipment may regain part of this loss in time. The coal industry must develop, test, promote, sell, and service such equipment. This can only be done through united effort.

Widespread use of the coal-fired combustion gas turbine locomotive will recover a part of the railroad fuel business, when this locomotive is put into production.

Promotional work, including better engineering of coal-burning equipment, is a very important part of the marketing of coal. The work of spreading the gospel of proper design of coal-burning plants, proper maintenance and supervision, and careful consideration of coal in all plans for expansion and in new plants must be doubled and redoubled. Coal salesmen must be experts in coal selling and application—not expert salesmen who can sell appliances, hairpins, or real estate with equal facility.

There must be full recognition of the present situation of the coal industry, of the trends which affect marketing and the courses open to change these trends. Responsible coal producers and sales companies must make a concerted effort to convince their responsible customers that a fair and adequate price for coal is a good investment. It cannot be done by hoping that someone else will do it.

Frontiers in Heat Extraction from the Combustion Gases of Coal

by Elmer R. Kaiser

COMBUSTION of coal and transfer of heat from flames and gases to boiler surfaces continue to be of great interest to engineers here and abroad. Numerous investigations have been in progress to improve furnace and boiler performance and economy. The importance of better understanding of the processes and opportunities for improvement is apparent when it is remembered that heat from at least 500 million tons of coal a year the world over is being transferred to boiler water at efficiencies ranging mostly between 50 and 90 pct. Even slight gains in efficiency, economy, and labor saving become very significant when multiplied by the enormous quantity of fuel consumed. Also the competitive position of the large coal, oil, and gas industries in satisfying the fuel consumers is greatly affected by the achievements made through technical progress with each fuel.

This paper is part of a continuing activity of Bituminous Coal Research, Inc., to extend the knowledge of coal utilization for steam generation and to seek promising directions for future research and development in cooperation with others. Particularly in the latter regard, numerous interviews were held during the last three years to seek the experience and advice of boiler and combustion-equipment manufacturers, electric-utility executives, and fuel engineers. A wealth of published information was also reviewed, which together with the interviews pointed to the advisability of further work on ash and sulphur control.

For the present purpose a number of factors important to efficient heat liberation and recovery have been grouped as follows: 1—combustion, temperatures, and rates of heat liberation; 2—radiation, convection, and furnace and boiler configuration; 3—sponge ash, slag, and hard-bonded deposits; 4—low-temperature deposits and corrosion (cooling flue gas below dew point and air-pollution control); 5—the limitations of coal cleaning and boiler size and cost as related to fuel characteristics; 6—future possibilities and conclusions.

The development of combustion apparatus for power boilers is progressing at a lively pace. There has been no letup in improvements in design of pulverized-coal-fired boilers, and there is a strong trend at present toward improving dry-bottom units. Spreader stokers with overfire jets and dust collectors as standard equipment are gaining favor. Less than 10 years in commercial use, cyclone burners are going into numerous installations here[1] and abroad.[2] Underfeed and traveling-grate stokers have long since been developed for heavy-duty operation, yet new developments in overfire jets and humidification of air blast have improved their performance. A water-cooled vibrating-grate stoker of German origin is being introduced into the United States and Canada.[3]

The primary objectives of an ideal coal combustion device are: capacity to burn the variety and sizes of coals likely to be economically available during the life of the unit; capacity to burn the coals automatically for a wide load range and rapid load fluctuations and to burn the coals completely to CO_2, H_2O, and SO_2, which means without smoke and cinders, or carbon in the refuse; capacity to control and discharge all the ash in final granular form without ash adhesion to walls or tubes, and without flue dust; minimum furnace volume; minimum labor and maintenance; low initial and operating cost.

Regardless of the method of burning, the gaseous products of coal combustion are N_2, CO_2, O_2, H_2O, and SO_2. By way of illustration, the coal analyses in Table I is assumed from an installation described by E. McCarthy.[4]

Table I. Products of Coal Combustion

Proximate Analysis, Pct		Ultimate Analysis, Pct	
Moisture	8.0	Moisture	8.0
Volatile matter	35.0	Carbon	67.5
Fixed carbon	47.0	Hydrogen	4.6
Ash	10.0	Nitrogen	1.4
		Oxygen	5.2
Total	100.0	Sulphur	3.3
		Ash	10.0
Btu per lb: 12,360		Total	100.0

When coal is burned with 20 pct excess air (theoretical air, 9.23 lb per lb of coal), the quantities of combustion gas shown in Table II are produced. In addition, the gases carry particles of fly ash, unconsumed cinders, soot particles, and small but significant amounts of vaporized oxides and sulphates of sodium, potassium, lithium, phosphorous, iron, and other metals. In recent years, germanium, one of the rare metals found in coal, has been shown to oxidize and vaporize at combustion temperatures and to be concentrated by recondensation at lower temperatures.[5]

Pulverized coal and cyclone flames[6] have peak temperatures of 3000° to 3500°F. Temperatures in fuel beds of large underfeed stokers reach maxima of 3000°F, sufficient to fuse almost any ash and to volatilize some of it. These peak temperatures are above the optimum necessary for rapid combustion, but they hasten heat transfer for ignition as well as boiler heat absorption. Furnace and gas temperatures increase with combustion air preheat. Low excess air has the same effect. Fine coal pulverization and highly turbulent combustion shorten the distance for fuel burnout, increase flame temperature, and speed up heat transfer.

Rates of combustion of pulverized coal exceeding 200,000 Btu per cu ft per hr have been demonstrated in atmospheric gas-turbine combusters,

E. R. KAISER, Member AIME, is Associate Director of Research, Bituminous Coal Research, Inc., 980 Kinnear Rd., Columbus, Ohio.

Discussion on this paper, TP 3737F, may be sent (2 copies) to AIME before May 31, 1954. Manuscript, Nov. 2, 1953. AIME-ASME Joint Fuels Conference, Chicago, October 1953.

vortex burners, and primary furnaces of boilers. The heat release rate in the cyclone furnace exceeds 400,000 Btu per cu ft per hr.[6] Similar heat releases can be obtained in fuel beds. With each of these types of combustion units it has been necessary to provide additional furnace volume to achieve 95 pct or higher completion of combustion. The hourly heat release per cubic feet of total furnace volume frequently ranges between 15,000 and 21,000 Btu for pulverized-coal-fired boilers.[4] Above 27,000 to 28,000 Btu per cu ft per hr the carbon loss may become excessive. Combustion rate is purposely low to insure adequate cooling of gases and fly ash by heat transfer to the furnace walls before the gases enter the convection banks.

With good mixing of fuel and air and adequate time for combustion, modern power boilers have reduced losses of unburned fuel of 1 pct or less, and excess air is only 20 to 25 pct.

Radiation, Convection, Furnace and Boiler Configuration

An excellent and recent review of the factors and mathematics of heat transfer in furnaces of water-tube boilers has been prepared by G. G. Thurlow.[7] The Research Committee on Furnace Performance Factors sponsored by the American Society of Mechanical Engineers has investigated not only the total heat absorption but the area heat-transfer rates in the furnace walls.[8-11]

Approximately half the heat liberated in a boiler is absorbed by the water-tubed furnace surfaces. Boiler furnaces do not operate under black-body conditions; the heat absorbed by radiation in a pulverized-coal-fired boiler is 0.6 or less that which would be absorbed under black-body conditions at the gas exit. The emissivity of particles, dust clouds, flames, and boiler surfaces has been studied by many investigators. Currently a cooperative program with international sponsorship is in progress in Holland to study flame radiation.[12]

Table II. Quantities of Combustion Gas Produced by Burning Coal With 20 Pct Air

Gas	Dry Volume, Pct	Lb Per Lb Coal	Lb Per Ton Coal	Volume of Gas at 330°F, Cu Ft Per Lb Coal
N_2	81.2	8.537	17,074	181.5
CO_2	15.0	2.475	4,950	33.5
O_2	3.6	0.428	856	8.0
H_2O		0.494	988	16.3
SO_2*	0.2	0.066	132	0.6
	100.0	12.000	24,000	239.9

* If all S were burned to SO_2, the dry volume percentage would be 0.27. Actually, some SO_3 and sulphates are formed.

In a modern pulverized-coal-fired boiler furnace with metal wall surfaces, heat is transferred from the flames to tube surfaces which are at temperatures up to 1200°F. Heat transfer is principally by radiation. Convection heat transfer is kept low, 3 to 14 pct, to reduce the rate of ash deposition on tubes and to prevent overheating of cleaned areas.

The design problem then becomes one of proportioning the furnace and locating the burners in the furnace walls to complete combustion, prevent direct impingement of gases and fly ash on the walls, and transfer enough heat to the water and steam to cool the products of combustion at least 150°F, and preferably 200°F, below the ash-softening temperature before the gases enter convection tube banks. It is well known that slag screens, divided furnaces, and tall furnaces accomplish these objectives. Also, installation of the convection tubes beyond a right-angle turn of the furnace exit gases prevents direct flame radiation on the tubes. This helps to prevent slag deposits on convection tubing.

A newer development coming into use is the tube curtain or platen, a partition of tangent water tubes extending into the furnace space to receive heat from both sides. Figs. 1 and 2 illustrate one application of tube curtains to cool furnace exit gases.[13] A number of boiler manufacturers apply platens for waterwalls, for convection surface, and for superheaters.

In an application, not illustrated, at the new Oak Creek Station of the Wisconsin Electric Power Co., three partition walls along the front wall form four semi-cells between down burners. Not only will the partition walls absorb heat from the pulverized-coal flames while combustion progresses, but the superheat and reheat temperatures can be controlled by differential firing into the semi-cells.[14] The boiler was designed for coal with a 2000°F ash-softening temperature.

The most critical condition is in the superheater and reheater tubing, which in some cases contains steam flowing at 1050°F. With steam inside the tube the heat transfer rate of 75,000 Btu an hr per sq ft of wall surface is an allowable maximum (300°F gradient through tube wall), while 40,000 Btu an hr per sq ft (150°F gradient through tube wall) is a desirable rate. Present practice for such boilers is 45,000 to 50,000 Btu per sq ft per hr, and future practice is predicted to be 35,000 to 40,000 Btu per sq ft per hr. Boilers with waterwalls may have heat absorption rates as high as 75,000 Btu per sq ft per hr.

Conventional boilers for high pressure operation use 3-in. diam tubes with thick walls which are more easily overstressed by heating than are tubes of smaller diameter and thinner walls. The development of boilers with pump-stimulated circulation of water has made practical the use of thinner tubes of smaller diameter.

The heat absorbed in the superheating and reheating sections of many units is 40 pct or more of the total heat input. With steam temperatures rising an average of 12°F a year, the trend is said to be inescapably toward more superheating and reheating in the furnace-wall tubes. The importance of radiant superheaters to supplement and balance convection superheaters is well known. Not all manufacturers agree on a trend toward radiant wall superheat and reheat.

Assuming clean tube surfaces at 1200°F, a heat transfer rate of 40,000 Btu per sq ft per hr by radiation only, and a combined radiation coefficient of 0.75, it is possible to calculate a mean temperature for the furnace by employing the Stephan-Bolzmann equation:[15]

$$q = k\left[\left(\frac{T_1}{1000}\right)^4 - \left(\frac{T_2}{1000}\right)^4\right]$$

where $q = 40{,}000$; $k = 1720 \times 0.75 = 1290$, a constant for the furnace conditions; and $T_2 = 1200 + 460 = 1660°$ R, outside tube temperature, then solving for $T_1 = 2490°R$ or 2030°F, which is the mean furnace temperature.

In other terms, the mean temperature of the surfaces and flame *seen* by a unit area of wall under consideration must be only about 2030°F if the heat transfer rate is to be 40,000 Btu per sq ft per hr. The center of the flame will be much hotter, but other walls seen by the unit area will be only 1200°F or less, with a combined equivalent of 2030°F.

When $q = 50{,}000$, $T_1 = 2140$°F
$q = 60{,}000$, $T_1 = 2250$°F
$q = 70{,}000$, $T_1 = 2350$°F
$q = 80{,}000$, $T_1 = 2430$°F
$q = 90{,}000$, $T_1 = 2510$°F
$q = 100{,}000$, $T_1 = 2580$°F

The present trend in heat absorption rates in dry-bottom furnaces is said to be downward in the electric-utility industry.

The lower furnace temperatures required in future practice will be obtained by lower heat absorption rates per square foot of furnace wall. Heat must be radiated from the burning particles and absorbed by the walls as the combustion of the particles proceeds. To illustrate the trend, design data are given in Table III for two plants of the Wisconsin Electric Power Co.[15]

Table III. Design Data for Heating Units, Wisconsin Electric Co.

Stations	Port Washington	Oak Creek
Year	1945	1953
Steam temperature, °F	900	1,000
Btu per sq ft per hr of furnace surface	85,000	65,000
Radiant heat absorption by superheater and reheater, Btu per sq ft per hr	42,000	32,000
Ash-softening temperature, °F	2,100	2,000

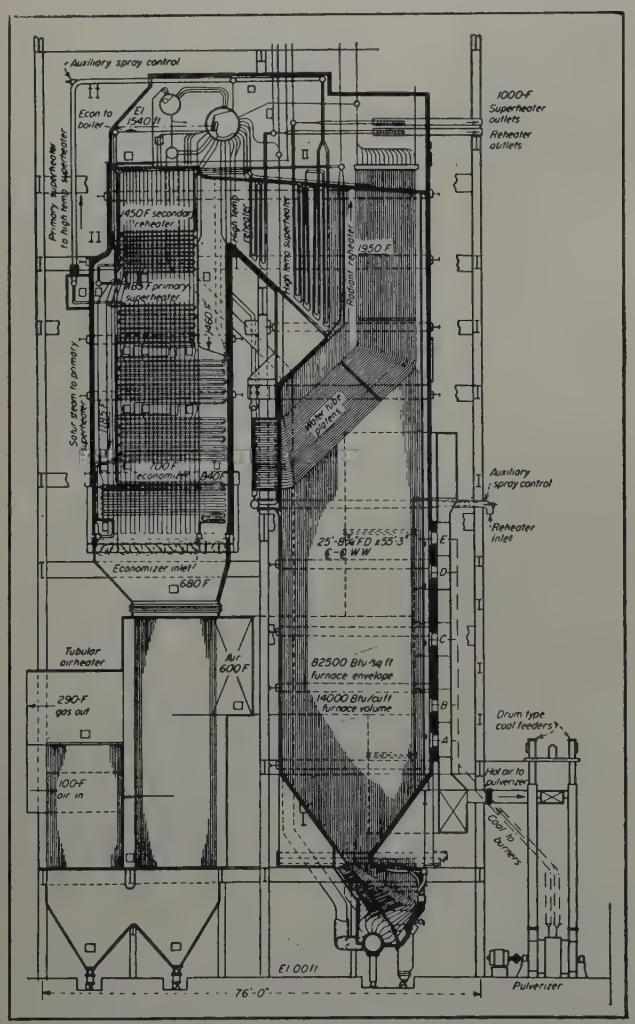


Fig. 1—A pulverized-coal-fired boiler with watertube curtains. (Courtesy *Power*).

Control of furnace temperature is also being achieved in some modern boilers by recirculation of flue gas and by adjustable burners for selectively using a part or all of the available furnace volume.

Information collected from several sources indicates a definite trend toward lower gas temperatures leaving the furnace to prevent spongelike deposits and slag on hotter tubes. Lower heat release and absorption rates are projected to assist in ash control and to prevent overheating of furnace tubes carrying high-temperature steam.

Sponge Ash, Slag, and Hard-Bonded Deposits

Sponge Ash and Slag: For most coal-fired boilers there is no serious problem with tight ash deposits on heat transfer surfaces. Where high ratings are carried and where ash-softening temperatures are low, the problem is more critical. Outstanding advances have been made in boiler equipment to prevent or control tight ash deposits.

Ash deposits on boiler surfaces insulate heat and in convection passes restrict gas flow. Since the outer surface of the slag is considerably hotter than the boiler tubes, heat flow is still further reduced. Reduced heat transfer causes higher flame temperature which in turn increases slag formation.

When coal is burned in pulverized form, all the ash is initially in suspension in the furnace gases, 50 to 70 pct or more of the ash being carried into the convection passes. With industrial stoker firing the quantity of solids suspended in the gases after combustion weigh between 5 and 40 pct of the weight of ash in the coal fired.[16] Only a minor fraction of dust leaving the furnaces with the gases is deposited on the boiler tubes, but the dust can form loose or tight coatings depending on temperatures and dust compositions.

Fig. 3 illustrates a cyclone-fired boiler, unique with respect to ash in that only 10 to 15 pct of the ash is carried out of the cyclone in suspension with the gases.[17]

The deposition of the dust on boiler tubes has been studied in detail by numerous investigators. The work done for the British Boiler Availability Committee is outstanding among the more recent studies of ash on boiler surfaces. The outside surface of the tubes is a thin film of oxide of the tube metal. The next layer on the tubes is a thin film of sulphates of iron, sodium, and potassium. These water-soluble compounds of low softening temperature form a base for the adhesion of particles of fly ash that reach the furnace tubes in dry or molten form. Molten ash particles sometimes impinge, fuse, and solidify on the tube metal or surface oxide.

The particles on the tube surface have a higher temperature than the tubes. When additional particles of dust deposit in successive layers, the temperatures rise and the particles gradually sinter together to form a surface that is both plastic and sticky. Surface tension and chemical action between the particles promote sintering and the evolution of gas respectively, with the result that a spongy mass is formed. The effect is most evident on the furnace walls and entrance to the convection zone. Elsewhere the temperatures are seldom high enough for vitrification, the deposit being dry and loose.

Delayed burnout of the coarser particles of pulverized coal, long flames, and near-flame impingement on wall surfaces increases rate of deposition because the resultant ash particles are hotter and stickier when they strike the tubes.

Ash particles vary in composition and softening temperature. Analyses of deposits of fly ash and sponge ash show average compositions of SiO_2, Al_2O_3, CaO, Fe_2O_3, and alkalis. A more detailed study of the compositions and fusion characteristics of the individual particles of mineral matter would probably reveal the way some act as cementing agents for others.

Once the buildup of ash deposits has begun, rate of deposition is accelerated. Deposits reduce the rate of heat absorption; hence gas temperature increases. Increased temperature promotes ash deposition and adherence and extends the zone of sintered deposits to surfaces farther along in the path of the gases.

Unless such deposits can be broken off by decrepitation, as by dropping load, or by lances and jets, the boiler must be taken off the line for cooling and manual cleaning.

Studies have been made of the behavior of ash after the layer has become thick and hot enough to flow. The viscosity of molten ash has been measured by P. Nicholls and W. T. Reid.[18] Messrs. Reid and P. Cohen reported on studies by the Bureau of Mines on factors affecting the thickness and flow characteristics of ash on furnace wall tubes.[19]

Mumford and Bice[20] have estimated that heat transfer to furnace tubes varies with the ash-deposit thickness as shown in Table IV.

Table IV. Slag-Ash Factors

Avg Thickness of Slag or Ash Deposit, In.	Factor
0 to 1/32	1.0
1/32 to ¼	0.7
¼ to 1	0.5
1 and over	0.3

Measurements made in a spreader-stoker-fired boiler, Fig. 4, show that dirty waterwalls can reduce heat absorption by 20 pct for the entire furnace.[9] In pulverized-coal firing, DeLorenzi states the furnace exit gases will be 200°F, or more, lower when the furnace walls are clean than when the walls are dirty.[21]

Clean tube surfaces show a period of immunity from deposits, sometimes up to six weeks, but in rare cases not at all. Tubes have been coated with aluminum, graphite, and lime with some beneficial effect for the life of the coating.

Boiler-tube corrosion from deposits is a serious problem and considerable progress has been made in alleviating it in the furnace. Information developed on corrosion of tubes will assist in an understanding of the nature of deposits next to the metal. A difference of opinion exists on whether the alkali sulphides cause tube wastage in reducing zones. Deposits of iron sulphide have caused trouble.

Ash may be classified into three categories: silicates, sulphides, and vaporized alkalis. The objective is to burn the sulphides and retain all the ash ingredients in the form of silicates. FeS and other sulphides are not miscible with slag.

It has been reported[22] that silicon sulphide (SiS) forms readily in a stoker fuel bed by reaction of

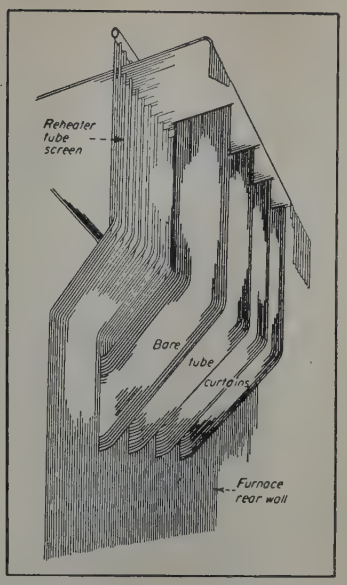


Fig. 2—Curtains of bare tubes in tangent contact. (Courtesy *Power*)

FeS_2 and SiO_2, especially if the oxygen content is low. The SiS sublimes at 1400°F and may deposit on surfaces below 1400°F. SiS is sticky at temperatures prevailing on superheater tubes and may cause ash particles to adhere. Oxygen and steam decompose it. Whether the same effect occurs in pulverized-coal firing is doubtful, but it should not be overlooked as a possibility.

The methods of removing slag from the furnace walls and the entrance to the convection passes are well known. Dropping the load temporarily has a marked effect. Soot blowers and wall deslaggers using either steam or air have been highly developed for the purpose. In troublesome zones not readily reached by soot blowers hand lances are employed.

By trial and error, users of coals from various sources determine which coals have ash characteristics suitable to their plants and loads. Unpredictable clinkering and slagging sometimes occur when mixtures of coals from different sources are consumed. Blowing loose ash off boiler tubes is not regarded as a problem. In general, users learn to adapt to the ash problem in older high-duty boilers, and select new boilers with more conservative furnace conditions. Nevertheless, it may be advisable to re-examine the limited knowledge of ash and develop 1—ash slagging tests for coals and 2—techniques for preventing adherent ash deposits.

Hard Bonded Deposits: The ash deposits most difficult to remove are the so-called bonded or alkali-matrix deposits that form on economizer, preheater, or superheater, and steaming-bank tubes. These deposits occur where the temperature of the flue gases is below the softening temperature of the ash. The fact that hard, bonded, enamel-like deposits that cannot be removed effectively by soot blowers can form on surfaces from 150° to 1100°F gives some idea of the wide scope of the problem. In cases where deposits form in superheaters, economizers, and air heaters, the condition of the scale indicates that chemical attack of the metal surface and of the ash itself takes place. Practical operating techniques to prevent these deposits are apparently not known, other than to increase excess air and to change coals. Fortunately the difficulty is limited to relatively few boilers and coals.

Chemical analyses of the deposits indicate the chief ingredient to be a sodium-potassium pyrosul-

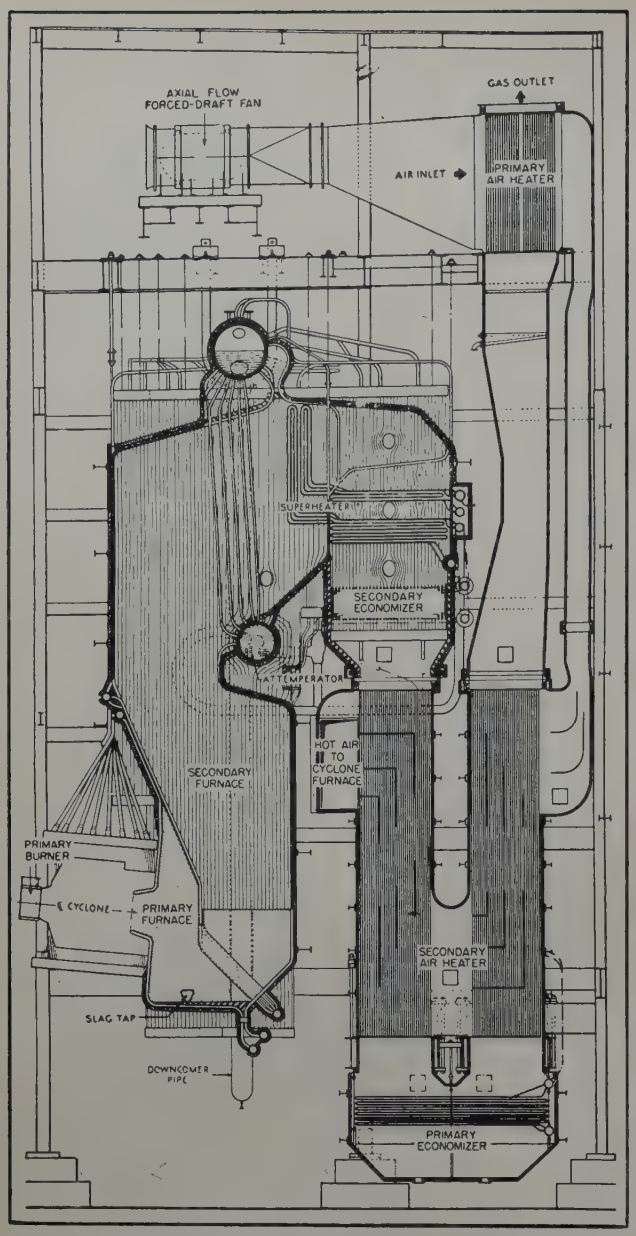


Fig. 3—A cyclone-furnace steam generator. (Courtesy *Power Generation*).

phate. The sulphate is probably the result of chemical reaction in the gas space of SO_3 with the alkali metals sublimed in the heat of the furnace. The sulphate then condenses on the tube where it forms a sticky surface to trap other particles of ash. The sulphate has a lower melting temperature than the silicates comprising the bulk of the ash.

The deposits are more severe in stoker-fired and cyclone-fired boilers than in pulverized-coal-fired boilers. The higher ratio of fly ash in the latter probably dilutes the sulphate deposit and acts, according to E. G. Bailey, like "flour on a pie board" in preventing bonding. The dry ash particles are also believed to form suspended nuclei for the condensation of alkali sulphates, rendering the sulphates harmless and carrying them into the dust collectors in dry form. Nevertheless, hard-bonded sulphate deposits occur in some pulverized-coal-fired boilers. Some plants in the United States have reported deposits containing as much as 40 pct sulphur trioxide even though the coal contained less than 1 pct sulphur.[22]

In the cooler zones of the steam generator, such as primary superheater and especially air preheater, the sulphate deposits are also accompanied by corrosion of the metal. High dew points of flue gas because of SO_3 are believed to be responsible for both corrosion and deposits. The sulphuric acid attacks the metal and traps and holds dust particles. Alumina is attacked to form aluminum sulphate, which is sticky and collects more fly ash or dust.

Sulphate-type deposits have been giving increasing troubles in the past few years and may be partly the result of the higher efficiencies and lower excess-air requirements of the modern installations. In a study by Barkley, Burdick, and Berk of the Bureau of Mines,[23] the deposits decreased as the CO_2 decreased. With 9 pct CO_2, only about one-third as much deposit was laid down as when 15 pct CO_2 was carried at about the same dew point. A boiler with economizer and air heater shows very little change in efficiency with minor changes in excess air. Enough excess air should be used to avoid troublesome deposits. Normal boiler design is based on 13.5 to 15 pct CO_2, varying with the coal.

Manual cleaning and water washing are employed to remove the deposits. The deposits weaken by water soaking, but much physical effort is required before the surfaces are clean. Regarding both sponge ash and bonded deposits, the British Boiler Availability Committee[24] recommended 1—that boiler surfaces be thoroughly cleaned of ash down to bare metal and 2—that ash buildup should not be permitted to increase draft loss by over 50 pct.

A specially designed pellet gun has been developed recently for removing slag deposits in very high-duty furnaces and in furnaces using particularly low-quality fuel.[25, 26] The gun is said to be effective against deposits originating in a cyclone furnace fired by a coal containing chlorides. Asphalt-asbestos pellets, ½-in. diam, are propelled by compressed air at rates of 50 to 250 pellets per sec to remove unusual deposits.

In Europe the Broman system of raining hard iron pellets over boiler tubing has been adapted to 90 boilers.[27] The pellets are recovered and re-used. Other developments involving tube vibration, ultrasonic agglomeration, and nozzle contour are currently being investigated.[26]

The ultimate solution to the problem of the bonded deposits will probably be found in chemistry. Much work remains to be done to determine the causes and cures. Sponge ash can be controlled better through boiler design than can the bonded deposit. A program of research of no small magnitude is indicated, including also the conditions of boiler operation that promote and prevent the deposits. The fact that pulverized-coal-fired boilers are relatively freer of these deposits than are stoker- and cyclone-fired boilers suggests that refractory dusts of fly ash might be added to the furnace atmospheres.

At one large British power station phosphatic deposits on heating surfaces, due to vaporization of phosphoric acid from the fuel bed, were overcome by introduction of pulverized coal above the stokers and recirculating part of the flue gas.[28]

Low-Temperature Deposits and Corrosion

Modern power boilers of high efficiency recover heat from boiler flue gases, first by means of economizers which preheat feedwater and then by preheaters of combustion air. The lowest practical

limit of cooling today is set by the temperature of the coldest preheater metal in contact with flue gas, i.e., where the cold inlet air enters the air heater. The metal temperature should be safely above the dew point of the flue gases.

At and below the dew point, sulphuric acid of high strength condenses and rapidly corrodes preheater steel. At the same time the acid forms sticky areas on which dust deposits build, ultimately clogging the gas passages. The sulphate of the preheater metal forms a deposit which contributes to the clogging and reduction of heat transfer. Because of the soluble nature of the deposits, periodic washing with hot water is effective.

The sulphuric acid (H_2SO_4) is produced when SO_3, usually under 0.02 pct, combines with water vapor in the flue gas. Even when only 1 or 2 pct of the sulphur in the coal burns to sulphur trioxide, the dew point is raised significantly. The dew point is not affected by SO_2 and only to a minor degree by the amount of water vapor normally present,[29] as shown by Fig. 5.

The high concentration of the sulphuric acid condensate is illustrated by reference to Fig. 6 from Johnstone.[30] For example, condensate produced between temperatures 300° and 150°F would have the approximate compositions between points A and B.

An electrical dew point meter has been developed,[31] but dust from the gases affects the dew point film and renders accurate determinations difficult.[32]

Many researchers have investigated the chemical mechanism and kinetics of SO_3 formation. The high temperatures in the furnace are unfavorable to SO_3 production, but between the furnace and preheater the iron oxide on boiler tubes may catalyze the reaction $2SO_2 + O_2 \rightarrow 2SO_3$ (Harlow). Ingredients in fly ash and the secondary combustion of small amounts of carbon monoxide (Whittingham) may also promote SO_3 formation. Gumz[33] has recently

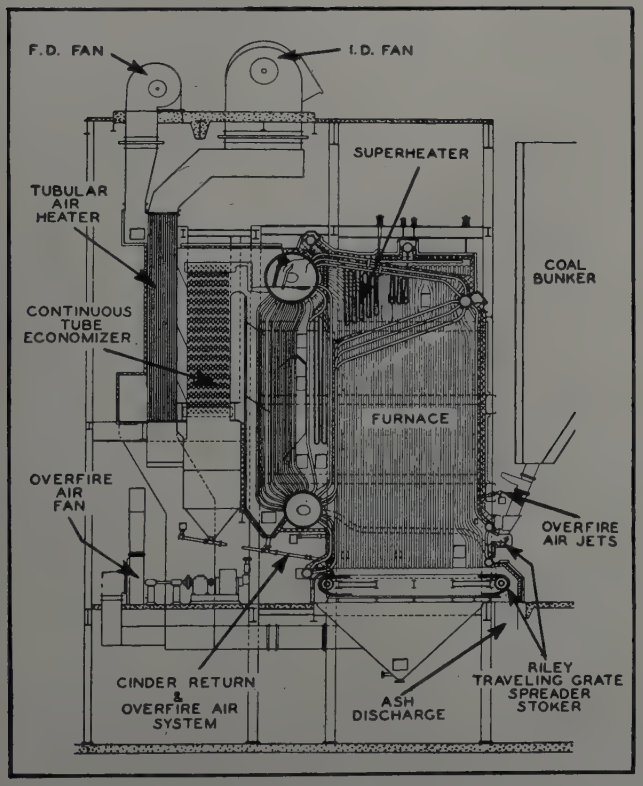


Fig. 4—A spreader-stoker-fired test boiler. (Courtesy American Society of Mechanical Engineers).

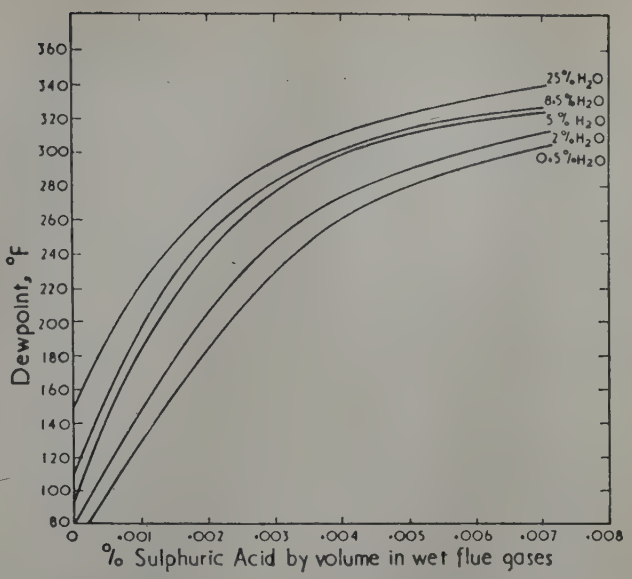


Fig. 5—Variation of dew point with H_2SO_4 content for gases having different water-vapor contents. (Courtesy British Coal Utilisation Research Association).

summarized the equilibrium data and pertinent references. Much more remains to be done to explain the chemical processes and to develop suitable controls. It is believed that corrosion and deposits can be lessened by reducing the sulphur in the fuel and by preventing the formation of SO_3.[34]

"To investigate and report on the problems of corrosion and fouling in equipment exposed to low-temperature flue gas" is the scope of the ASME Research Committee on Low Temperature Flue Gas Corrosion and Deposits, which was organized in 1951 with John F. Barkley as chairman.

A recent Bureau of Mines report[34] on corrosion and deposits in regenerative air preheaters illustrates the type of corrosion experienced and the comparative rates of corrosion of the engineering metals and alloys. Cast iron, alloy and plain steels, copper, aluminum, bronze, and monel metal were attacked. It was concluded that all metals that form sulphates cannot be used for air-preheater plates without corrosion and eventual plate failure.

Similar results have been found with tubular recuperative air heaters.[35] Berk[36] has reviewed British experience, and Henning and Rögener have reported German difficulties with economizers.[32]

Several steps have been taken toward practical solutions of the problem. In economizers, the corrosion is prevented by elevating the feedwater temperature. In air preheaters, the inlet air is first warmed by recirculating with it part of the preheated air. The air warming might also be done with indirect heat from exhaust steam or in a cheaper expendable air heater.

An automatic means for regulating the air-inlet temperatures to preheaters so as to prevent dew point conditions would be highly desirable. A practical and reliable dew point indicator for dusty flue gas would be the first step toward this objective.

Addition of dolomite ($CaCO_3$-$MgCO_3$) to a 2.75-pct-sulphur fuel oil by the Florida Power Corp. is reported to have lowered the dew point from 250° to 275°F down to 150°F.[37] Loose calcium sulphate ($CaSO_4$) was formed. The effect of dolomite on air-heater corrosion has not yet been determined, either for oil or coal. The effect on SO_3 in flue gas of

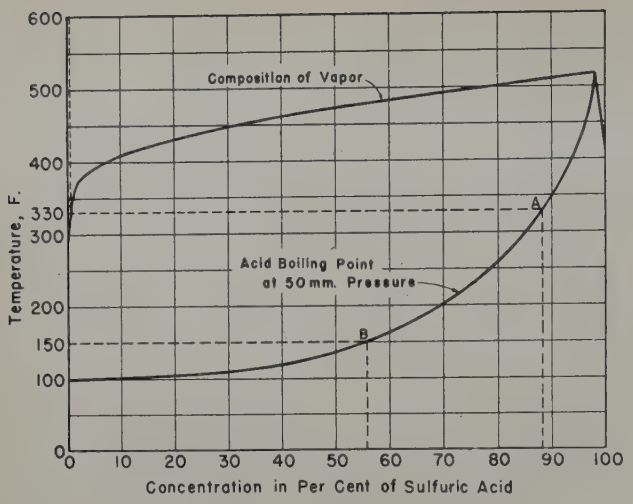


Fig. 6—Boiling point-vapor composition curve for sulphuric acid solutions at 50 mm Hg pressure. (Courtesy H. F. Johnstone).

the natural lime in coal ash or limestone additive to coal is also apparently unknown.

Substitution of special glass tubing for metal in the condensation region of air heaters is being tried, as is a method for periodic replacement of short lengths of corrodible tubing.[35] However, the clogging of the tubing by acid-trapped dust would likely still be a problem. This leads to the suggestion that high-efficiency dust collectors be installed ahead of the air heaters.

Where spreader stokers are used, the cinders carried through the air heater would help to scour off deposits. Where that is the case, dust collectors may well be located after the air heaters.

Cooling Flue Gas Below Dew Point: Future opportunities and possible developments are not being overlooked. An interesting and possibly promising line of attack would be deliberately to produce a maximum of SO_3 with a view toward condensing it out with the ample quantities of water vapor already present in flue gas. The air preheater to condense the acid would have to be of acid-proof construction. Instead of discharging the flue gas at 330°F as in the example,[4] heat might be extracted down to an arbitrary temperature of 150°F. Table V lists the sources and amounts of recoverable heat for this installation. Only a small fraction of the water vapor in the flue gas would be condensed.

Table V. Recoverable Heat in Flue Gases Between 330°F and 150°F

Source of Heat	Lb Per Lb Coal	Btu Released Per Lb Coal	Btu in Coal, Pct
SO_2 to SO_3 reaction	0.082	42	0.34
H_2O sensible heat	0.494	43	0.35
H_2O latent heat of condensation	0.044	44	0.36
$H_2O + SO_3 = H_2SO_4$ (80 pct strength, approx.)	0.127	31	0.25
Dry gases	11.506	496	4.00
Total		656	5.30

The hypothetical change in the heat balance for the boiler is given in Table VI.

Even if only half the sulphur in the coal were converted to SO_3 and the gases were cooled to 150°F, additional heat recovery would be 4.83 pct.

The weight of raw acid may be sufficient to wash away the dust trapped, especially if the acid were recirculated for that purpose.[38]

If the SO_3 in normal flue-gas concentration were removed from the flue gas by a suitable dry reagent and the gases were then cooled to 150°F, no condensation of moisture would occur and no acid would be formed. The added heat recovery would be 4.35 pct. Where flue gas is presently cooled to 250° to 275°F, the opportunity for gain would be proportionally less. Some large boilers now have average operating efficiencies of 89 to 90 pct.

These and other possibilities are under study to increase heat recovery. The increased heat transfer surface for air preheating will add to the investment of the boiler, but the heat recovery should be sufficient to establish the economic balance at a lower flue-gas temperature than is now practical.

Air Pollution Control: No study of this kind would today be complete without considering the effect of combustion methods and heat-transfer equipment on air pollution. Smoke and soot have fortunately been reduced to low levels by better combustion. Fly-ash emission is being brought under control by dust collectors and precipitators, but more needs to be done. Economical removal of sulphur oxides from flue gas has been investigated for years by Johnstone in the United States and by others abroad.[39] Flue-gas washing has been practiced in a few plants,[40] but the disposal of the effluent water created serious problems.

By further cooling of the flue gases to recover heat, it may be possible to combine the removal of at least some of the sulphur oxides and more of the flue dust.

Table VI. Heat Balances in Pct of Btu in Coal

Stack Gas Temperature	330°F		150°F
Heat absorbed by steam-generating unit	85.69		90.09
Heat loss in dry chimney gases	5.47		1.47
due to moisture in air	0.14	4.64	
due to moisture in coal	0.43		3.93
due to combustion of hydrogen	4.07		
due to unconsumed carbon	2.00		2.00
due to radiation and unaccounted	2.20		1.61
	100.00		100.00

An interesting sidelight is the beneficial effect of SO_3 on the collection efficiency of electrical precipitators[41] through lowered electrical resistivity of the fly ash in the presence of low percentages of SO_3.

Experimentation with new principles and chemical processes which combine recovery of heat and air-pollutants may lead to an economical answer.

Limitations of Coal Cleaning: The misbehavior of ash and sulphur in boilers naturally suggests consideration of the advantages and economics of coal cleaning. As is well known, the sulphur and ash contents of coals can be reduced by density separation of particles of higher and lower ash contents near a selected specific gravity. Modern coal preparation plants improve coal quality by reducing the ash and sulphur percentages, and by producing a more uniform product. The effect on the ash-softening temperature varies. Depending on the coal, cleaning may raise or lower the ash-softening temperature.

Better coal preparation has been the trend and many users can attest to the benefits. However, there are definite physical and economic limitations to coal cleaning. The present cost of coal preparation for steam generation, which generally ranges between 35¢ and 75¢ a ton of product coal, permits

removal of only the free impurities, plus possibly heat drying the finer sizes of washed coal.

A number of coal producers have investigated the cost of further reducing ash contents beyond present practical limits. The process involves progressively crushing the coal to release particles high in ash content and cleaning at lower specific gravities. The yield of final product coal is reduced by the more intensive cleaning (separation), as more tonnage and Btu's are rejected to gob. Two examples of confidential origin may be cited in Table VII.

Table VII. Cost Studies of Raw and Washed Coals at Two Mines

Eastern Bituminous Coal		Midwestern Bituminous Coal	
Ash Content, Dry Basis, Pct	Cost* Per Ton of Product	Ash Content, Dry Basis, Pct	Cost* Per Ton of Product
15.5 raw	$3.50**	16.5 raw	$3.50**
8.5	4.40	6.5	4.75
7.5	4.75	6.0	4.80
6.5	5.25	5.5	4.90
5.5	6.05	5.0	5.20
		4.5	5.70
		4.0	6.90
		3.5	8.50

* Includes washing and loss of coal.
** Arbitrary.

The rapidly rising cost per ton of cleaned coal is evident for both coals. The difference in cost between the two coals reflects the greater difficulty of cleaning this particular eastern coal as compared with the midwestern coal.

The reduction of sulphur, iron, and alkali contents of the coal is even more difficult and more impractical by methods known today than is the reduction of ash. Free marcasite and pyrite (FeS_2) particles of macroscopic size (density = 5) are readily removed in washing because of their high density as compared with coal (1.3 to 1.5). However, part of the sulphur and iron is thoroughly distributed in coal as sulphate and as finely disseminated pyrite particles of microscopic size. Sodium and potassium are present as chlorides. They are distributed throughout the coal and the shaly matter.

Because of the technical and economic limitations on coal cleaning by known processes, boiler and combustion-equipment designs have been developed to cope with the ash and sulphur problems. In this way the most favorable overall cost balance can be struck on the basis of cost of steam.

Beyond the cost of Btu's delivered, the economic value of well-prepared industrial steam coal for large modern steam generators is a subject for evaluation by each coal consumer and coal source. Ash increases the coal tonnage to be handled and pulverized. Boiler operating labor and maintenance increase with ash content of the coal. When ash deposits and slagging force a reduction in boiler load or shutdown, the loss could have paid for much coal preparation, particularly if the boiler load must be shifted to older and less efficient peak-load equipment. Complete information is lacking, for even one boiler plant, to make possible a comparison of cost of operation and maintenance for two coals of different ash contents and different characteristics.

Boiler Size and Cost as Related to Fuel Characteristics: Boilers are designed to fit a fuel specification. Boilers designed for coals of high ash-softening temperature can be more compact than those designed for coals of low ash-softening temperature. Larger furnaces are required for the latter coals to reduce the temperature of the gases entering the first convection bank 150°F below the ash-softening temperature. The tube spacing must also be larger in the first convection zone to prevent bridging of ash deposits.

The design and selection of boilers is a teamwork job between boiler manufacturer and utility purchaser. Some select more conservatively than others. After the boiler is installed, the operating crew must learn the characteristics of the boiler and the control of combustion and ash.

The magnitude of the difference in cost between boilers for different fuels was investigated with the boiler manufacturers. The purpose of the inquiries was to determine the cost of liberal boiler sizing as one way of reducing the tight ash deposits on boiler surfaces.

During an interview, one manufacturer prepared estimates for a 900,000-lb-an-hr steam generator, 1850 psi, 1000°F. Included were an outdoor-type boiler, superheater, economizer, air heater, soot blowers, pulverizer, coal feeders, dust collector, but not feedwater pumps, fans, or walkways. The figures are summarized in Table VIII.

The increased price for a boiler to consume coals of low ash-softening temperature was 7 pct. The increase in cost was primarily due to the increased size of furnace and convection passes and extra heat-exchange surfaces required. The estimate for an "ashless" coal-fired boiler is based on experience with natural gas, except that the cost of coal feeders and pulverizer was included, but not dust collector or soot blowers.

Table VIII. Relative Prices of Boilers and Auxiliaries

Type of Bituminous Coal	Price of Unit, Pct	Relative Price, Pct
"Ashless"	100	
Ash of AST 2750°F	131	100
Ash of AST 2100°F	140	107

In 1944 the Fairmont Coal Bureau[42] published a similar analysis made by another boiler manufacturer, see Table IX.

Table IX. Relative Prices of Boilers and Auxiliaries, Fairmont Coal Bureau

400,000 lb steam per hr Efficiency: 87 pct Pulverizer firing, bituminous coal	950 psi, 850°F Capacity factor: 85 pct	
	For Coal of AST 2550°F	For Coal of AST 2070°F
Furnace:		
Width	21 ft 10 in.	21 ft 10 in
Height	34 ft 6 in.	42 ft 6 in.
Depth	20 ft 1½ in.	24 ft 0 in.
Volume, cu ft	19,900	28,600
Additional furnace volume cost		$22,000
Additional building volume		30,000 cu ft
Additional building cost		$15,000
Total additional cost		$37,000
Total cost of the boiler and auxiliaries	$368,000	$390,000*
Pct increase in cost of boiler only		6

* 60 pct of 1950 price.

The increased price of 6 pct for a boiler to burn low-AST coal compares closely with that of 7 pct obtained independently in the first example.

The additional investment in the larger boiler must be amortized by savings in cost of Btu's in the lower-fusion coal. The effect on the price of coal can be illustrated with conservative example:

Assumptions:
Boiler of 425,000 lb per hr steaming capacity.
Cost installed: $1,000,000 for high-AST coal.
Additional cost of $100,000 for low-AST coal.
Coal: 11,500 Btu per lb.
Evaporation: 9.42 lb water per lb coal.
Load factor of 80 pct, availability 95 pct.
Avg load = 425,000 x 0.80 = 340,000 lb an hr.
Annual coal consumption = 340,000 x 0.95 x 8760/(9.42 x 2000) = 150,000 tons.
Amortization of increment investment over 6 yr: $100,000/6 = $16,700 a year.
Amortization rate = $16,700/150,000 = $0.11 per ton of coal.

The additional cost per ton of coal is so low that it is now common practice for utilities to increase their fuel flexibility• by installing boilers designed for a wide range of coals. The increment cost in a larger boiler is less than any known preparation or treatment of a low-AST coal to render it suitable for the smaller boiler. A boiler manufacturer advised that the boiler should be made to suit the worst conditions, that the furnace should be built large enough, and that gas temperatures are lower with a clean furnace.

As indicated earlier, other ash and sulphur factors must also be considered. Operating labor increases with the quantity of ash handled. A larger tonnage of high ash coal must be unloaded, stored, and conveyed for equal steam output. Increased maintenance on coal and ash conveyors, higher cost of ash removal, more frequent soot blowing, and higher pulverizer wear are among the costs that must be considered.

Future Possibilities and Conclusions

1—The designs of modern large power boilers and combustion devices are highly advanced for thermal efficiency and for capacity to cope with ash in coal.

2—Further research on ash behavior and slag prevention would be desirable. Improved soot blowers, pellet guns, tube vibrators, and operating techniques now under development will also assist in keeping boiler tubes clean. The manifold advantage of tube curtains in their various forms will help reduce furnace temperatures and adherent deposits.

3—The role of non-sulphate ash dust in condensing the alkali sulphates and rendering them nonadherent to tubes should be investigated with a view to wider practical application.

4—Cooling of flue gases to 150°F is a worthy objective provided that corrosion and deposits in air preheaters can be prevented. Each 35° to 45°F reduction in flue-gas temperature increases boiler efficiency by 1 pct.

5—Several hypothetical possibilities exist for the control of SO_3 in flue gas: SO_3 formation might be prevented by techniques to be developed, but the likelihood is slight. Normal amounts of SO_3 in flue gas might be absorbed on basic dusts ahead of or in the air preheater. SO_3 could be condensed out with water vapor by cooling of gases in acid-proof preheaters.

6—SO_3 now assists in reducing air pollution from power plants by reducing electrical resistivity of fly ash in electrical precipitators. In future air preheaters of acid-proof construction, the sulphuric acid produced by SO_3 and water vapor may be useful in trapping flue dust. The acid could be recirculated if necessary to wash the dust off the surfaces. It may become advantageous to convert as much as possible of the sulphur to SO_3.

7—Economic and technical opportunities for solving the ash and sulphur problems with coal appear to be more promising at present through combustion and utilization in power boilers than through supercleaning of coals beyond the present economic limits.

References

[1] H. C. Schroeder and R. J. Strasser: Station Design With Cyclone-Fired Steam Generators. *Trans.* ASME (May 1952) **74**, pp. 573-578.

[2] O. Rosahl: Einflusse des Einbindungsgrades, der Staubrückführung und der Entstaubung auf den Staubaustrag. *Mitteilungen der Vereinigung der Grosskesselbesitzer* (March 1953) **23**, pp. 1-14.

[3] Schuettelrost Steinmueller: U.S. licensee, American Engineering Co., Philadelphia. Vibra Grate stoker.

[4] Edmund McCarthy: Pulverized Coal Firing. *Utilization* (April 1952) pp. 43-47.

[5] T. Stadnichenko, K. J. Murata, P. Zubovic, and E. L. Hufschmidt: Concentration of Germanium in the Ash of American Coals. A Progress Report. U.S. Geol. Survey *Circ. 272* (1953).

[6] Frank X. Gilg: The Cyclone Furnace. *Power* (April 1950) pp. 64-70.

[7] G. G. Thurlow: Heat Transfer in the Furnaces of Water-Tube Boilers. BCURA Monthly *Bulletin* (May 1953) **17**, No. 5, pp. 173-199.

[8] F. G. Feeley, Jr. and E. C. Miller: Furnace Heat Absorption in a Spreader-Stoker-Fired Steam Generator. Part II, Paper 52-A-142. ASME Annual Meeting, 1952.

[9] J. W. Myers and R. C. Corey: Furnace Heat Absorption in a Spreader-Stoker-Fired Steam Generator. Part I. Paper 52-A-153. ASME Annual Meeting, 1952.

[10] J. W. Myers and R. C. Corey: Furnace Heat Absorption in Pulverized-Coal-Fired Steam Generator. *Trans.* ASME (May 1951) **73**, pp. 419-455.

[11] F. G. Ely and N. H. Twyman: Variation in Heat Absorption as Shown by Measurement of Surface Temperature of Exposed Side of Furnace Tubes. Part II. *Trans.* ASME (May 1951) **73**, pp. 419-455.

[12] Flame Radiation Research Joint Committee (Secretary P. A. H. Elliot, London): Report of 1949 Trials at IJmuiden. *Journal of the Institute of Fuel,* November 1951, January 1952, and later.

[13] Max H. Kuhner: Furnace Design of Large Steam Generators. *Power* (August 1952) pp. 78-79.

[14] O. J. Stallkamp: Design Features of the Oak Creek Station. *Proc.* American Power Conference (1952) **14**, pp. 234-240.

[15] M. K. Drewry: Wisconsin Electric Power Co., Milwaukee, Wis.

[16] J. F. Barkley: Current Developments in Reducing Fly Ash Emission from Chimneys. *Proc.* 33rd Annual Convention, Smoke Prevention Association (1939).

[17] V. L. Stone and I. L. Wade: Operating Experiences With Cyclone-Fired Steam Generators. *Mechanical Engineering* (May 1952) **74**, pp. 359-368.

[18] P. Nicholls and W. T. Reid: Viscosity of Coal-Ash Slags. *Trans.* ASME (1940) pp. 141-153.

[19] W. T. Reid and P. Cohen: Factors Affecting the Thickness of Coal-Ash Slag on Furnace Wall Tubes. *Trans.* ASME (November 1944) **66**, pp. 685-690.

[20] A. R. Mumford and G. W. Bice: An Investigation of the Variation in Heat Absorption in a Pulverized-Coal-Fired Water-Cooled Steam-Boiler Furnace. Part IV. Comparison and Correlation of the Result of Furnace Heat Absorption Investigations. *Trans.* ASME (July 1948) **70**, pp. 604-607.

[21] Otto DeLorenzi: Influence of Low-Quality Coal on Design and Operation of Pulverized-Coal-Fired Units. Paper 52-F-48, ASME Fall Meeting (1952).

[22] Deposits on Gas Side of Steam Generating Units. Edison Electric Institute (February 1951).

[23] J. F. Barkley, L. R. Burdick, and A. A. Berk: Test Data on Gas-Side Sulfate-Type Deposits on Tubes

Beyond the Boiler Furnace. ASME *Trans.* (February 1948) **70**, pp. 81-87.

[24] The External Fouling and Corrosion of Boiler Plant (1940-1951); Second Interim Report, same (1946-1951); Summary of Published Papers, February 1953. The Boiler Availability Committee, 8 Waterloo Place, Pall Mall, London, S.W. 1.

[25] W. F. Cantieri: Recent Developments in the Use of Pellets for the Removal of Slag Deposits in Boiler Furnaces, ASME Annual Meeting 1952; briefed in *Power Notes*, Diamond Power Specialty Corp. (May-June 1953) pp. 6-7.

[26] Lyle B. Schueler: Boiler Cleaning Systems—Principles and Practices, Paper 53-SA-24, ASME Semi-Annual Meeting (1953).

[27] H. Theobalt: Rauchgasseitige Kesselreinigung. *Brennstoff-Wärmekraft* (1951) Bd. 3, p. 194. Also BWK (September 1953) p. 329.

[28] Facts and Figures. *Combustion* (September 1953) p. 57.

[29] G. Whittingham: Some Effects of Moisture on the Combustion of Coal. Monthly Bulletin (June 1953) British Coal Utilisation Research Association.

[30] H. F. Johnstone: An Electrical Method for the Determination of the Dew Point of Flue Gases. *Circ. 20*, Engineering Experiment Station, University of Illinois (1929) p. 18.

[31] P. F. Corbett, D. Flint, and R. F. Littlejohn. Developments in the BCURA Dew Point Meter. British Coal Utilisation Research Association, *Inf. Circ. 55*, 1951, pp. 1-19.

[32] F. Henning and H. Rogener: Ergebnisse von Taupunktmessung an Hochdruckkesseln. BWK, August 1953, pp. 269-272.

[33] W. Gumz: Brennstoffschwefel und Rauchgastaupunkt. BWK (August 1953) pp. 264-269.

[34] J. F. Barkley, Hilmer Karlsson, A. A. Berk, C. F. Stack, and L. R. Burdick: Corrosion and Deposits in Regenerative Air Preheaters. U. S. Bur. Mines *R. I. 4996* (August 1953).

[35] E. F. Rothemich and G. Parmakian: Tubular Air Heater Problems. Paper 52-A-124, ASME Annual Meeting (1952).

[36] A. A. Berk: Work in Great Britain on Corrosion and Deposits in Air Preheaters. *Mechanical Engineering* (July 1953) pp. 545-546.

[37] J. B. McIlroy, E. J. Holler, and R. B. Lee: The Application of Additives to Fuel Oil and Their Use in Steam-Generating Units. Paper 52-A-160, ASME Annual Meeting 1952.

[38] British Patent 688,658. The Air Preheater Corp. Applic. date Dec. 30, 1949.

[39] S. R. Craxford, A. Poll, and W. J. S. Walker: Recovery of Sulfur from Flue Gas by the Use of Ammonia. *Journal of the Institute of Fuel* (London) (March 1953) pp. 357-358, Disc. pp. 358-362.

[40] R. L. Rees: The Removal of Oxides of Sulfur from Flue Gases. *Journal of the Institute of Fuel* (London) (March 1953) pp. 350-357.

[41] H. J. White: Electrical Resistivity of Fly Ash (May 1953) Research Corp., Bound Brook, N. J.

[42] Better Engineering for New Plants, Fairmont Coal Bureau, New York City, *Bull. No. 5* (September 1944).

A Physical Explanation of the Empirical Laws of Comminution

by D. R. Walker and M. C. Shaw

The laws of Kick and Rittinger are explained as functions of particle size with metal cutting theory. Comminution is shown to be basically the same process as metal grinding. The machine shop type of grinding operation is used to study mineral crushing. Evidence indicates that plastic flow occurs in comminution.

THE laws of comminution of Kick and Rittinger have been debated for many years. Certain data obtained from ball mill and drop tests are found to be in approximate agreement with Rittinger's law while other data would seem to support Kick's law. In all these tests the energy required to produce a wide variety of particle sizes has been studied. Surface area has also been measured with considerable difficulty, at first because it was thought that the energy associated with crushing was that required to create new surface, later because of precedent and because a more fundamental variable was unknown.

Tests made with the machine shop type of grinding process show that normally brittle materials that are usually crushed can behave like the more ductile metals. Brittleness is then a relative property largely dependent on specimen size and stress state. The machine grinding operation offers a convenient means for studying the comminution of materials in which the important size variable retains its identity. The energy required to grind a certain volume of material is shown to be strongly dependent on size. Kick's law is found to hold very well for particles below 1 micron in size, while Rittinger's law holds approximately in the comminution of larger particles. While a physical origin of Kick's law is established in terms of the theoretical strength of materials having a perfect lattice structure, there is found to be no physical basis for Rittinger's law.

Because of its industrial importance, comminution has commanded considerable technical attention and has been the subject of continuous study for nearly a century. A number of empirical rules have been proposed to make it possible for grinding energies to be predicted. Of these so-called laws of comminution those bearing the names of Kick and Rittinger have been most widely applied and debated.

D. R. WALKER and M. C. SHAW are, respectively, Assistant Professor and Head, Machine Tool Division, Department of Mechanical Engineering, Massachusetts Institute of Technology.

Discussion on this paper, TP 3677B, may be sent (2 copies) to AIME before May 31, 1954. Manuscript, Sept. 5, 1952. Chicago Meeting, September 1952.

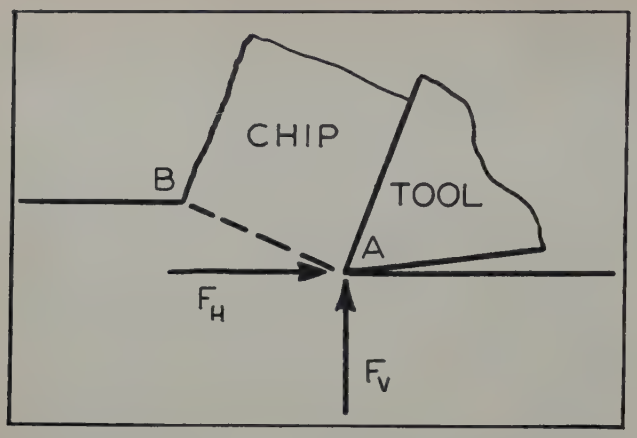


Fig. 1—Schematic representation of metal cutting operation.

Kick's law is based upon a critical strain energy concept. It states that the energy required to fracture a specimen is directly proportional to its volume and independent of the number or size of particles into which it is reduced. Stated algebraically this is

$$U = K_1 V \qquad [1]$$

where U is the energy to which the volume V must be subjected to cause fracture and K_1 is a proportionality constant.

The law of Rittinger is based upon the prediction that the energy required to crush a substance is that needed to produce the new surface generated. Or,

$$U = K_2 (A_2 - A_1) \qquad [2]$$

where U is the energy required to crush a material when the change in area involved is $(A_2 - A_1)$, and K_2 is a proportionality constant.

Investigators using Rittinger's law have found that crushing efficiencies are generally extremely low. Some data of Martin[1] who crushed quartz in an 18 x 18-in. tube ball mill using 1-in. diam balls are shown in Table I.

Table I. Representative Ball Mill Data

Energy Consumed, U Ft-Lb	New Area Produced $(A_2\text{-}A_1)$ Ft²	$U/A_2\text{-}A_1 = K_2$ Ft-Lb Per Sq Ft
243,375	3,971	61.3
470,250	7,852	59.9
699,190	11,170	62.6
892,346	14,941	59.7
1,097,300	17,899	61 3
		Average 60.9 ft-lb per sq ft = 5.07 in-lb per sq in.

The data of Table I are seen to be in good qualitative agreement with Rittinger's law, but in poor agreement with Kick's law. However, the surface energy of quartz is known to be about 1000 ergs per sq cm, which is 0.0057 in-lb per sq in. in English units. When this value is compared with the mean value of K_2 in Table I, it is found that only 0.112 pct of the energy associated with the process is being used to supply the necessary surface energy. Thus from Rittinger's point of view the efficiency of Martin's ball mill is but 0.112 pct, which is remarkably low for any engineering process.

Much of the literature in the field of comminution is found to consist of arguments supporting or rejecting one or the other of the two laws which have been mentioned here. Some investigators have found that their data tends to obey Kick's law, while others have gathered evidence which they use emphatically to defend Rittinger's law. Haultain[2] in 1923 and more recently Bond[3] have suggested that the actual law lies half way between those of Kick and Rittinger.

Experimental work in the field of comminution has been subject to several difficulties. In ball-milling experiments the total energy consumed in crushing a charge from one mean particle size to another has been measured. This energy is the energy required to crush particles of a wide variety of sizes. From such data it is not possible to determine precisely the energy required to crush particles of any one size. The chief variable studied has been the surface area, determined in the past by elaborate screening analyses and more precisely in recent years by means of gas adsorption and permeability techniques.[4] In view of the very small percentage of the energy of comminution associated with the development of new surface, it would appear that entirely too much emphasis has been placed on surface area in previous investigations.

Drop tests have been adopted in some studies to improve the experimental precision. However, interpretation of the data is still made difficult by the variety of particle sizes that result in such tests. It would appear that a substantially different experimental approach is needed to develop further the concepts of comminution.

In addition to comminution there is a second grinding process practiced on a wide industrial scale. This is the type of grinding done in the machine shop, where the objective is to produce a smooth, dimensionally accurate surface. Unlike the powders produced in comminution, the small chips formed in machine grinding are not of value and hence are usually discarded. Even though the objectives in comminution and machine grinding are diametrically opposed, there is considerable similarity between the two processes.

While it might be argued that comminution is concerned with materials normally considered brittle, whereas machine grinding involves metals having appreciably greater ductility, it will be shown later in this paper that this difference is actually more apparent than real. Since the intention is to apply to the comminution process techniques developed in the study of metal cutting, it may be well to review briefly the manner in which a metal behaves

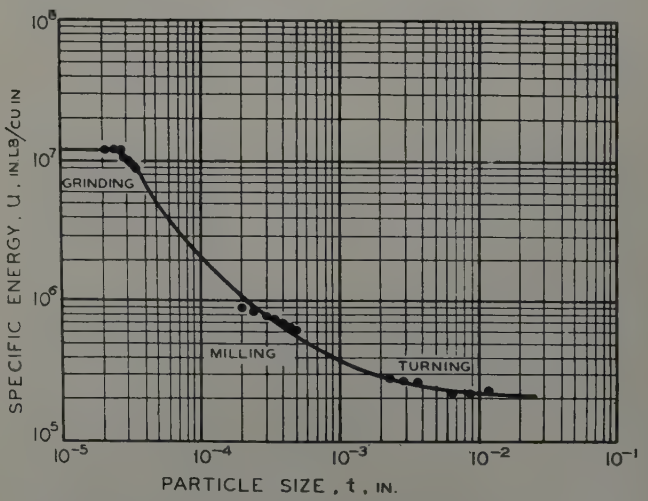


Fig. 2—Variation of specific energy with particle size for SAE 1112 steel.

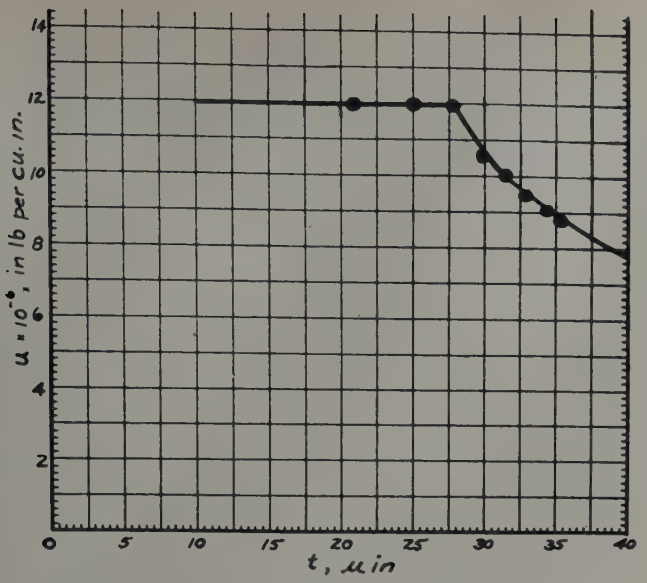


Fig. 3—Variation of specific energy with particle size in grinding for SAE 1112 steel.

when it is cut, before continuing the discussion of comminution.

Metal cutting is a shear process in which a chip is caused to flow plastically when a tool is advanced across the workpiece. The action is as shown in Fig. 1. The material below line *AB* (the shear plane) is plastically undeformed, while the material in the chip above *AB* is deformed uniformly. All the shear strain is confined to a very narrow zone extending along *AB*. As the metal crosses line *AB* it is subjected to a relatively large and rapidly applied shearing strain.

The calculation of the energy consumed per unit volume of metal cut or specific energy is one of the parameters which has proved to be very important in metal cutting studies.[5] When the depth of cut, t, taken by a tool, is decreased, the specific energy, u, is found to increase. The curve shown in Fig. 2 which covers an extremely large range of size was recently obtained[6] by use of a tensile test and three different cutting operations: turning, very high speed milling, and grinding. The material in all these tests was SAE 1112 steel.

The grinding end of Fig. 2 is shown to a larger scale in Fig. 3. Here it is evident that the energy per unit volume remains constant as the depth of cut is increased to a point, but then proceeds to decrease with further increase in depth of cut. The depth of cut or layer thickness corresponding to the break in the curve is about 27 micro in. or ¾ of a micron. It is of interest to this discussion to consider the cause for the sudden break in the energy curve of Fig. 3.

When an energy balance is made for a cutting operation it is found that practically all the energy is associated with the shear and friction processes. As in comminution, the surface energy involved in the generation of new surface is negligible, and energy associated with change in velocity of the metal as it crosses the shear plane also can be neglected.

$$u = u_s + u_f \qquad [3]$$

where u is the total energy per unit volume of metal cut, u_s is the shear energy per unit volume, and u_f the energy per unit volume required to overcome friction on the tool face. It has been shown in a recent paper[6] that in fine grinding operations u_s and u_f are about equal, while the shear strain in the chip is about 3. Since in plastic flow the shear energy per unit volume (u_s) is equal to the product of the average shear stress and the shear strain, then

$$u \cong 2\,u_s \cong 2\tau\gamma \qquad [4]$$

Since γ is approximately 3

$$\tau \cong \frac{u}{6} \qquad [5]$$

The value of u for the flat portion of Fig. 3 is seen to be about $12\text{x}10^6$ in.-lb per cu in. and the corresponding value of shear stress is thus found to be about $2\text{x}10^6$ psi from Eq. 5.

In Fig. 4 a true stress-strain tensile curve is shown for the material of Figs. 2 and 3, together with the stress-strain points for the cutting tests. In plotting the cutting points shear stress and shear strain were converted to normal stress and strain coordinates by use of the maximum shear theory. From Fig. 4 it is evident that the stresses required for cutting increase significantly as the depth of cut is decreased. The large variation in specific energy that was observed to occur with specimen size, Fig. 2, is thus seen to be due to a corresponding change in the flow stress of the material with specimen size. While the flow stress in shear is but about 50,000 psi for the tensile test specimen of 0.505 in. diam, the shear flow stress in grinding is found to be about $2\text{x}10^6$ psi when the particle size is less than 27 micro in.

For a long time physicists have held that metals should exhibit much higher values of flow stress and strength than they actually do. The strength of a metal specimen is found to be $G/2\pi$, where G is the elastic shear modulus of the metal, in one approximate calculation[6] based upon the forces required to slide one layer of atoms over another in a material having a perfect lattice structure. For steel, G is about $11.5\text{x}10^6$ psi, and hence the theoretical shear strength of a steel specimen with no lattice imperfections should be about $1.8\text{x}10^6$ psi, i.e., $\frac{11.5\text{x}10^6}{2\pi}$.

This value is seen to be in good agreement with the $2\text{x}10^6$ psi observed for the maximum shear stress in fine grinding. It would thus appear that the specific energy (and hence the shear stress) remains constant as long as the depth of the layer removed is less than that corresponding to the spacing of the inhomogeneities that are found in all metals. At a depth of cut of about ¾ microns the probability of encountering an inhomogeneity becomes noticeable and from this

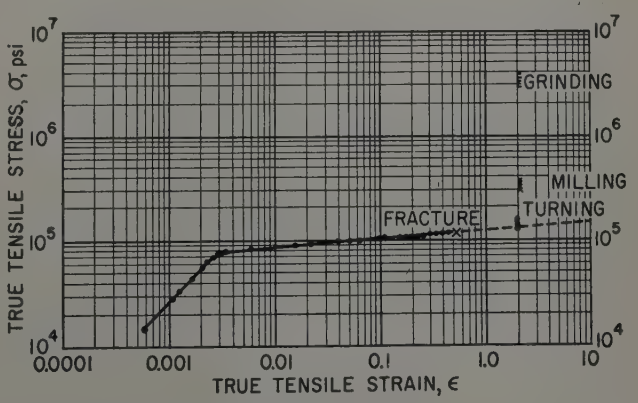


Fig. 4—True stress-strain tensile curve for SAE 1112 steel specimen, with cutting data added.

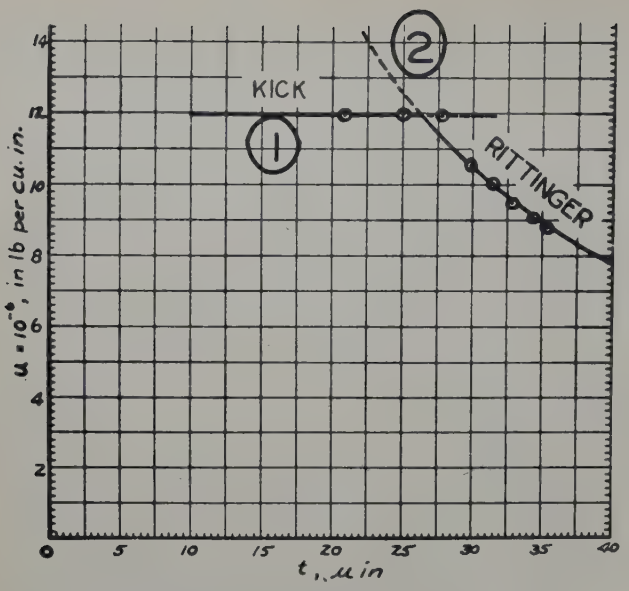


Fig. 5—Relation between grinding data of Fig. 3 and equations of Kick and Rittinger.

point on the flow stress of the material decreases markedly with an increase of the depth of the layer removed and hence the probability of finding an inhomogeneity.

From x-ray diffraction studies on metals, the size of the crystallites within which no lattice imperfections are found has been estimated to be about 10^{-4} cm (1 micron). This value is seen to be in good agreement with the thickness of the layer at which the effect of inhomogeneities become noticeable in metal cutting. Furthermore, the energy per unit volume is found to be independent of the hardness of the metal cut when the depth of cut is below the critical value of about ¾ micron, but it is found to be very important for larger cuts. This observation is in agreement with the foregoing picture. If the theoretical strength of the metal is $G/2\pi$, it should be unchanged by hardness, since G is known to be independent of hardness. Thus the shear stress and energy per unit volume in a cutting operation with a chip size less than about 27 micro in. should be independent of hardness.

From the brief review of recent metal cutting research that has been presented here it is evident that the size of the particle removed is an extremely important variable. The shear stress associated with the removal of a particle is found to increase significantly with decreased particle size, owing to the decreased probability of finding weakening inhomogeneities with decreased size. When a point is reached, in the reduction of size, where essentially no inhomogeneities are encountered, the strength of the metal remains constant with further reduction in size. The shear energy per unit volume is found to reflect directly changes occurring in shear strength.

The laws of Kick and Rittinger will now be interpreted in terms of the metal-cutting data that have been presented. If both sides of Eq. 1 are divided by the volume of the particle, then

$$u = \frac{U}{V} = K_1 \qquad [6]$$

which states that the energy per unit volume is a constant. This is seen to be in agreement with the horizontal portion of Fig. 2, and hence Kick's law may be said to be in agreement with metal cutting data when the depth of the layer removed is below the critical value.

In expressing Rittinger's equation in terms of specific energy it will first be observed that no change in volume accompanies any plastic deformation. Hence,

$$V_1 = V_2 = V \qquad [7]$$

If both sides of Eq. 2 are then divided by V,

$$u = \frac{U}{V} = K_2\left(\frac{A_2 - A_1}{V}\right) = K_2\left(\frac{A_2 - A_1}{V_2 - V_1}\right) \qquad [8]$$

This is seen to be the definite integral of

$$\frac{du}{d\left(\frac{A}{V}\right)} = K_2 \qquad [9]$$

When this equation is integrated

$$u = K_2\,\frac{A}{V} + u_o \qquad [10]$$

is obtained where u_o is the integration constant. However, as indicated in Eq. 11,

$$\frac{A}{V} = \frac{1}{t} \qquad [11]$$

V varies directly with the product of A and t where t is a mean thickness of the layer removed. Hence, from Eq. 10 and 11

$$u - u_o = \frac{K_2}{t} \qquad [12]$$

In Fig. 5 the data of Fig. 3 are shown replotted, and curve 2 is drawn in accordance with Eq. 11, the constants being:

$u_o = 200{,}000$ in.-lb per cu in.
$K_2 = 300$ in.-lb per sq in.

This curve is seen to be in good agreement with the grinding data. However, when this same curve, 2, is plotted with all the cutting data, see Fig. 6, the agreement is not quite as good. The milling data would appear to be in better agreement with curve 3 having constants

$u_o = 416{,}000$ in.-lb per cu in.
$K_2 = 103$ in.-lb per sq in.

From this comparison of the laws of Kick and

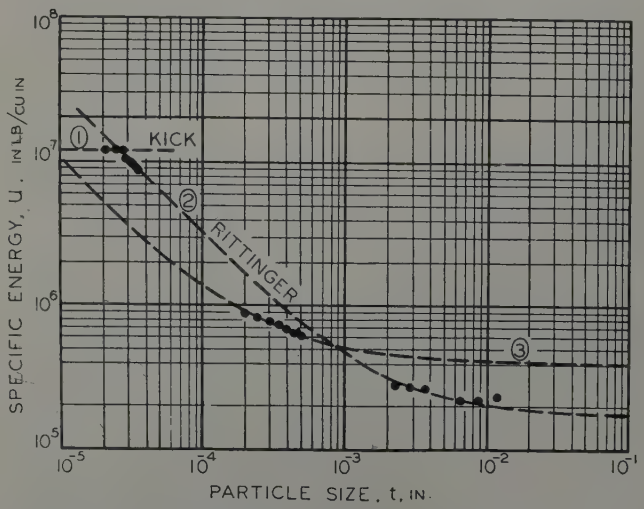


Fig. 6—Relation between cutting data of Fig. 2 and equations of Kick and Rittinger.

Rittinger with metal cutting data it is evident that Kick's expression

$$U = 11.95\ V, \text{ in.-lb} \qquad [13]$$

is in good agreement with cutting data when the depth of cut is less than about ¾ micron, while Rittinger's expression

$$U = 300\ \Delta A, \text{ in.-lb} \qquad [14]$$

is in good agreement with the grinding data, while the expression

$$U = 103\ \Delta A \qquad [15]$$

is in better agreement with the milling data than Eq. 14. However, these expressions are not in such good agreement with the experimental data for the reasons originally given by their authors. Kick's law applies because the material exhibits full theoretical strength and not because of any correctness of a strain energy concept. Rittinger's law holds despite the fact that the surface energy on which it is based is completely negligible. It is only because of the fact that a simple functional relationship exists between the size of a particle and its surface area that the Rittinger expression can be written in terms of area. The particle size is obviously the important variable, and Rittinger's expression should preferably be written

$$u - 200{,}000 = \frac{300}{t} \text{ in.-lb. per cu in.} \qquad [16]$$

in as much as the emphasis is then upon the physically important variable, t, rather than the physically unimportant variable, ΔA, as in Eq. 13.

Mineral Grinding

To investigate any differences that might be observed between the machine grinding of ductile metals and materials normally considered brittle, tests similar to those already described for metals were performed on minerals. The detailed test procedure is to be found in reference.[6] Some grinding tests were run using a 46-grit 8-structure aluminum oxide wheel, while others were made using a 24-grit aluminum oxide abrasive belt. The belt made it possible to remove chips of greater size than could be handled by the wheel.

Energy curves for marble, gypsum, and talc are given in Figs. 7 to 9. There is a striking resemblance between these data and the data for SAE 1112 steel given in Fig. 6. There is a region, *1*, for which the energy per unit volume is constant, followed by a region where the energy per unit volume decreases with depth of cut. It was found in each case that a curve in agreement with Eq. 12 (Rittinger's law) could be passed through the grinding-wheel data or the belt-grinding data, but that the same curve did not hold for both of these regions. This was also found to be the case with the metal, as may be seen in Fig. 6. This result simply means that while Rit-

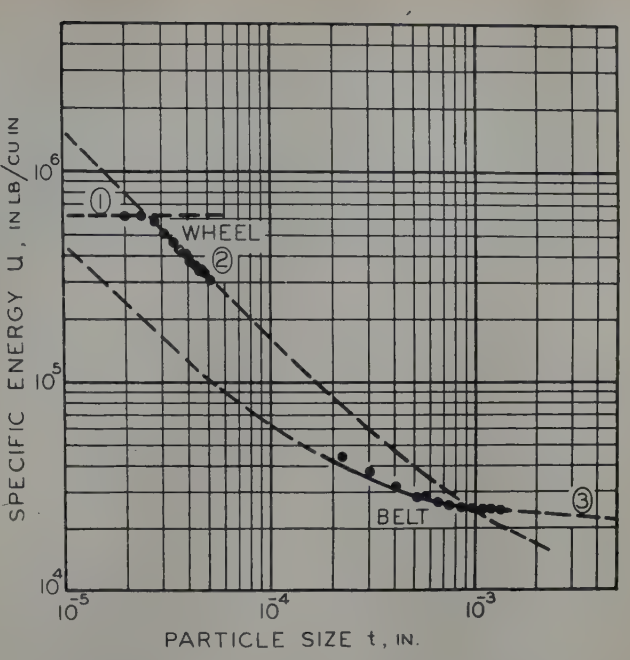


Fig. 8—Variation of specific energy with the particle size for gypsum.

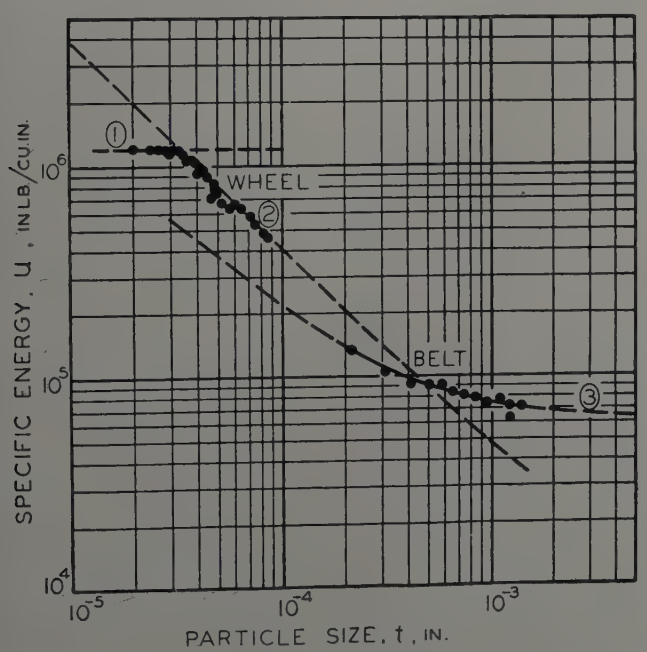


Fig. 7—Variation of specific energy with the particle size for marble.

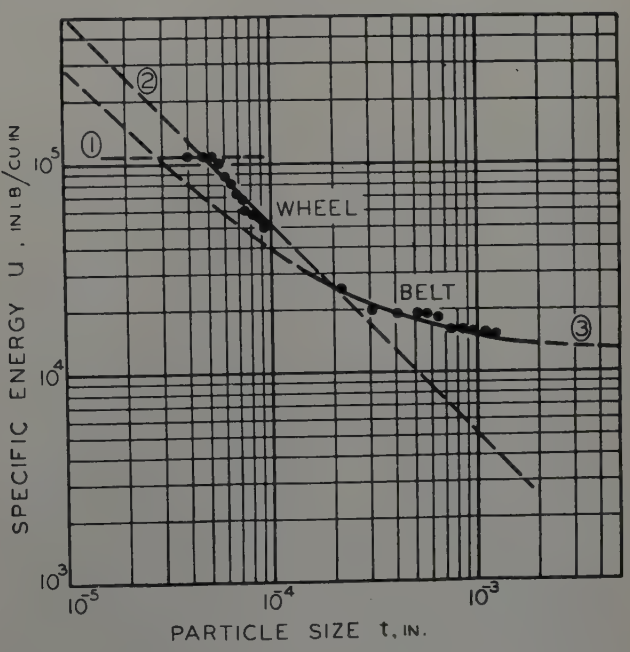


Fig. 9—Variation of specific energy with the particle size for talc.

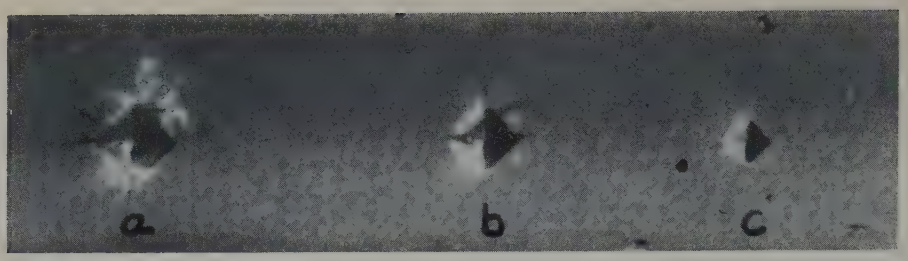


Fig. 10—Impressions made in marble with a diamond pyramidal indentor under loads of (a) 2.15 g, (b) 10 g, and (c) 5 g X 1200.

tinger's law holds quite well over a limited range of size it must only be regarded as a first approximation when it is applied over a wider range. The constants for energy equations 6 and 12 are summarized in Table II for the tests of Figs. 7 to 9. The values of K_2 are seen to decrease while those of u_o increase in a systematic way along the range from the small particle size characteristic of wheel grinding to the larger particle size of belt grinding. It would appear that the Rittinger expression, Eq. 12, does not predict quite as much increase in energy with decreasing particle size as is actually observed. This is evident in Figs. 7 to 9 where curve *3* is seen to be below curve *2* for small depths but above curve *2* for large depths.

The constant K_2 may be interpreted as the energy required to generate a unit quantity of new surface. This quantity is seen to vary not only with the material ground but also with the size of the particles removed. For example, the value of K_2 for marble is approximately 39.3 in.-lb per sq in. for the size range extending from 30 to 90 μ in., while K_2 equals about 16.2 in.-lb. per sq in. for the size range from 200 to 2000 μ in. Since the surface energy for marble is about 0.006 in.-lb. per sq in., it is again evident that the surface energy represents a very small percentage of the energy required to generate a unit of new surface.

Table II. Energy Constants for Mineral Grinding

Material	K_1 In.-Lb Per Cu In.	Critical Size μ In.	Region 2 u_o In.-Lb Per Cu In.	Region 2 K_2 In.-Lb Per Sq In.	Region 3 u_o In.-Lb Per Cu In.	Region 3 K_2 In.-Lb Per Sq In.
Marble	1.20×10^6	33	8500	39.3	59000	16.2
Gypsum	0.62×10^6	25	9100	15.3	20600	4.3
Talc	0.11×10^6	50	0	5.0	12200	2.76

Some investigators have reported that Kick's law holds best when particles of large size result from comminution. Figs. 7 to 9 show that the curves of specific energy vs particle size tend to become flat in the region of large particle size. Rittinger's law and Kick's law will thus give similar results in this size region. The tendency toward constant specific energy for comminution of large particles is to be expected since the larger the particle size becomes, the more the particle behaves like the material in bulk, and the specific energy of fracture thus tends towards that for fracturing the bulk material. This loss of size effect when the specimen becomes quite large is also observed in metals. Tensile tests made with metals show no dependence upon the size of the specimen as long as the specimen diameter is larger than about 0.1 in.

Such materials as marble are normally considered to fracture without undergoing previous plastic strain. Materials that behave in this way are said to be brittle. If marble is ground in the region in which Kick's law holds (u = constant), the shear stress developed at rupture would be the theoretical value, $G/2\pi$, where G is the shear modulus. If the material were perfectly brittle it would behave elastically to the point of rupture and from Hooke's law ($\tau = G\gamma$) the shear energy per unit volume involved would be

$$u_s = \frac{1}{2}\tau\gamma = \frac{1}{2}\frac{\tau^2}{G} \qquad [16]$$

Substituting $G/2\pi$ for τ,

$$u_s = \frac{G}{8\pi^2} \qquad [17]$$

Now, the shear modulus for marble is about 3.15×10^6 psi and hence the theoretical strength and elastic shear energy per unit volume would be as follows:

$$\tau = \frac{G}{2\pi} = 0.5 \times 10^6 \text{ psi}$$

$$u_s = \frac{G}{8\pi^2} = 0.04 \times 10^6 \text{ psi}$$

When this specific elastic shear energy is compared with the total energy per unit volume observed in the Kick's law region for marble (1.2×10^6), it would appear that

$$\frac{1.2 - 0.04}{1.2} \times 100 = 97 \text{ pct}$$

of total energy was consumed in friction. Since in the grinding of steel only about 50 pct of the energy input is associated with friction, the 97 pct figure would seem to be far too high, and hence it is suspected that marble is not really brittle under the grinding conditions investigated here, but rather flows plastically to a considerable extent before rupturing. This plastic flow would tend to increase the amount of shear energy in the process and hence lower the percentage of friction energy.

Plastic flow in grinding or any type of cutting process can be carried to far greater strains without rupture than in the case of a tensile specimen. For example, the strain to rupture in a true-stress-strain tensile test is about 0.5, while the corresponding strain in cutting the same material will be 2.0 or more without rupture, Fig. 4. The reason for these large strains lies in the fact that a large normal stress is present on the shear plane in cutting, and this normal stress increases the rupture stress. As previously mentioned, all materials contain a great many minute cracks and other inhomogeneities, and a normal stress on a shear plane prevents these cracks from growing to cause rupture. A material that contains large flaws will rupture at a lower stress level than one that contains smaller flaws.

Therefore, the strength of a small specimen will exceed that for a large specimen, since the probability of finding a flaw of a given size will be less in the small specimen. There are thus two important reasons why materials in grinding should be capable of withstanding much higher stresses without fracture than in bulk: 1—because of their small size and 2—because of the presence of a large normal stress on the shear plane.

When these two elements are present, materials that normally behave in a brittle manner in bulk may exhibit considerable ductility. For example, marble is usually considered to be a brittle substance, since it exhibits low tensile strength in bulk and essentially no plastic deformation before rupture. However, it is possible to cause extensive plastic strain in marble if the specimen is small and the pressure high. In Fig. 10 three impressions are shown that were made in a polished marble surface by a diamond pyramidal indentor. A different load was used in each case. In the first and second impressions a good plastic impression of the diamond is seen but there is some evidence of cracks extending from the corners of the impression. In the case of the third impression plastic flow has occurred, but no cracks are resolved at a magnification of 1200 x. This example illustrates that brittleness is a quantity that has meaning only when the size of the specimen is specified. Furthermore, it might be said that a material will behave in a brittle manner when its size is such that it contains cracks of sufficient size to preclude any measurable plastic deformation before fracture. Glasses, whether in the annealed or strained state, contain a large number of imperfections which vary widely in size; hence glasses are brittle over a wide range of size.

The Vickers hardness values (load in kilograms divided by projected area of impression in square millimeters) corresponding to the impressions of Fig. 10 are as follows:

Load, Gm	Vicker's Hardness, Kg/mm²
15	400
10	417
5	475

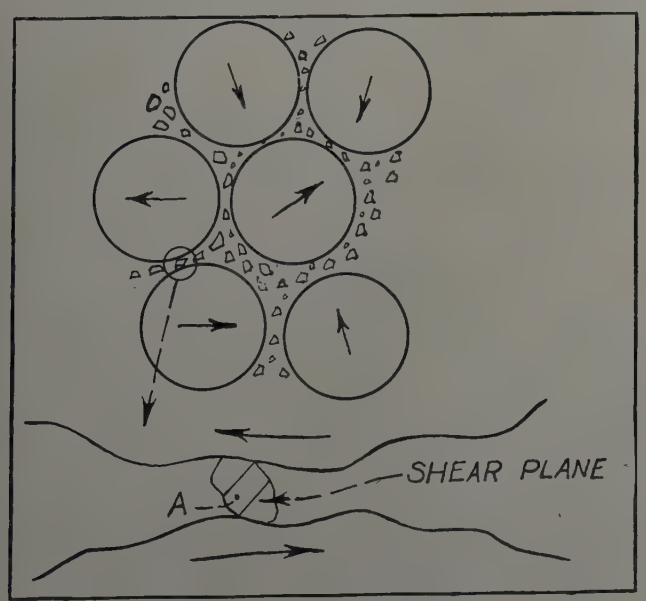


Fig. 11—Schematic representation of a particle being crushed in a ball milling operation.

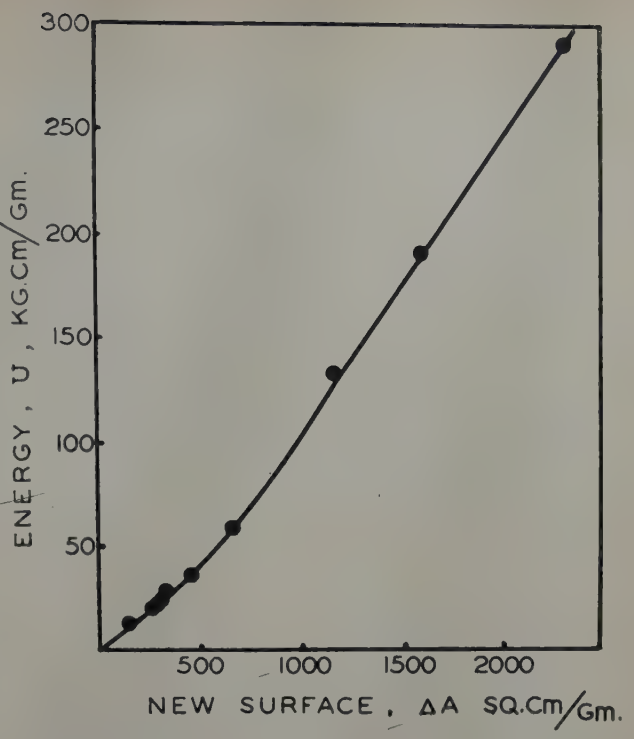


Fig. 12—Energy vs change in surface area, after Piret.

This trend of increased hardness is another indication of the size effect, Eq. 12. The diamond indentor is geometrically similar for all depths of penetration; thus if the material were homogeneous in its properties, the same hardness value should be obtained for all impression depths and hence for all loads. However, the plastic flow stress of the marble is seen to increase as the volume of the material deformed is decreased, indicating that the material is not homogeneous with regard to size.

In all tests reported here, the critical particle thickness varied from 25 to 50 micro in. X-ray diffraction and electron microscope studies have revealed the mean spacings of the inhomogeneities in metals to be about 1 micron. Rosenthal and Kaufman[7] have recently investigated the size of a perfect crystallite of Yule marble. The marble was first plastically deformed from 15 to 25 pct while a hydrostatic pressure of 10,000 atmospheres was acting to prevent rupture. The deformed specimen was then crushed and sized, and the X-ray line broadening of specimens of different particle size determined. Line broadening was found to disappear in specimens below 1 micron in size. These experiments show that the line broadening is due to residual stresses between crystallites. They also establish the size of the particle within which no plastic deformation occurred at 1 micron. In the grinding data given in Fig. 7 the critical depth of cut below which theoretical values of strength were achieved was found to be about 50 μ in or about 1.25 micron. This value is seen to be in good agreement with the results of Rosenthal and Kaufman for marble.

The close similarity between machine grinding of metals and the comminution of materials normally considered brittle is shown by their tendency to follow the same energy laws. On closer examination the difference in the ductility of the materials normally ground in these two processes is not found to be real. The usually brittle substances that are normally crushed are really quite ductile in the size range in which comminution occurs. A schematic

diagram of a particle being crushed in a ball mill is shown in Fig. 11. Since the particle will be small compared with the size of the balls, it can readily be forced to shear when compressed between the balls. If the particle is small enough so that it contains no inhomogeneities it should be expected to shear along the planes indicated and to exhibit its theoretical strength. The energy relation for this type of action will follow the law of Kick. If an inhomogeneity is present in the particle as at A, rupture will occur on a plane passing through this point and a stress level below the theoretical value will be obtained.

Piret and his associates have reported[4] test results in which crystalline quartz was crushed in a carefully controlled drop test. The resulting area was determined by the gas adsorption method. Representative values of surface area are shown plotted against the corresponding crushing energy in Fig. 12. According to Rittinger's law, Eq. 2, this should be a straight line. However, the slope of the curve

$$\frac{dV}{d(\Delta A)} = K_2$$

is seen to increase as the particle size is decreased, i.e., as the area, ΔA, generated increases. This observed increase in K_2 with decreased particle size is in qualitative agreement with Table II for the minerals and the curves of Fig. 6 for the metal specimen. This is further evidenced in support of the observation that Rittinger's law does not provide a large enough size effect. However, the results of Piret et al shown in Fig. 12 somewhat mask the actual deviation from Rittinger's law, in as much as the values plotted represent the result of several blows in a drop test. The values of energy plotted are thus the mean values for the range of particles produced in the several blows. Owing to the very strong size effect that has been shown to exist in crushing, a truer energy picture is obtained when the energies considered refer to a single particle size rather than to a range of particle sizes.

Concluding Remarks

The importance of the particle size upon the energy required to crush a material has not been recognized in previous comminution studies. Instead, the physically unimportant surface area has been adopted as the variable against which to correlate energy consumption. Furthermore, comminution experiments in the past have been concerned with energies associated with a range of particle sizes rather than with particles of one size. This has led to considerable controversy concerning the relationship between energy and surface area.

In the machine grinding operation, the energy consumed in particle formation can be studied under conditions that provide particles of essentially constant size. When data thus obtained is analyzed it is found that Kick's law holds for very fine grinding, i.e., for particles less than about 1 micron. On the other hand, Rittinger's law is found to be in agreement with experimental data only when the range of particle size involved is relatively small. When the data considered covers an appreciable range of particle size, Rittinger's law predicts too small a size effect. Kick's law naturally arises from the concept of a theoretical strength in the region of extremely fine particle size, and from consideration of properties of the material in bulk when in the region of extremely coarse particle size. However, although Rittinger's law qualitatively holds quite well over limited size ranges, there is no apparent physical basis for Rittinger's prediction that is in agreement with current concepts.

In this paper it has been indicated that appreciable plastic deformation is associated with the grinding of materials ordinarily considered brittle. For this reason there is little difference between machine grinding and other comminution processes such as ball milling. It would appear that the machine grinding technique described here offers a precise means for evaluating the grinding characteristics of various materials.

Acknowledgments

This study of the comminution process was supported by grant No. 202 of the National Science Foundation and is an outgrowth of an investigation of the grinding of metals sponsored by the Carborundum Co. The authors wish to acknowledge the help of Professor W. H. Dennen of the Geology Department in providing the mineral specimens and much useful information. Mr. W. E. Littman of the Metallurgy Department was kind enough to help in the preparation of the indentation photomicrographs, and Professor H. Rush Spedden, formerly of the Metallurgy Department, provided important literature and information. It should be mentioned that the idea that a connection might exist between machine grinding and ball milling was originally expressed by Professor T. K. Sherwood of the Chemical Engineering Department in a conversation in the authors' laboratory two years ago.

References

[1] G. Martin: Some Recent Research in the Science of Fine Grinding Done by the British Portland Cement Research Association Between 1923-1925. *Journal of the Society of Chemical Industry* (1926) **45**, p. 1601.

[2] H. E. T. Haultain: A Contribution to the Kick vs Rittinger Dispute. *Trans.* AIME (1923) **69**, p. 183.

[3] Fred C. Bond: The Third Theory of Comminution. *Trans.* AIME (1952) **193**, p. 484.

[4] J. M. S. Kwong, J. T. Adams, J. F. Johnson, and E. L. Piret: Energy—New Surface Relationship in the Crushing of Solids. *Chemical Engineering Progress* (1949) **45**, pp. 508, 655, 708.

[5] M. E. Merchant: Mechanics of the Metal Cutting Process. *Journal of Applied Physics* (1945) **16**, pp. 267, 318.

[6] W. R. Backer, E. R. Marshall, and M. C. Shaw: The Size Effect in Metal Cutting. *Trans.* ASME (1952) **74**, p. 61.

[7] D. Rosenthal and M. Kaufman: X-Ray Line Broadening of Plastically Deformed Marble. *Journal of Applied Physics* (1952) **23**, p. 600.

Additional References

H. Stadler: Grading Analyses and Their Application. *Trans.* Institution of Mining and Metallurgy (1910) **19**, p. 478.

A. O. Gates: Kick Vs. Rittinger: An Experimental Investigation of Rock Crushing Performed at Purdue University. *Trans.* AIME (1915) **52**, p. 875.

A. M. Gaudin, J. Gross, and S. R. Zimmerly: The So-Called Kick Law Applied to Fine Grinding. *Mining and Metallurgy* (1929) **10**, p. 447.

M. C. Shaw: A Yield Criterion for Ductile Metals Based Upon Atomic Structure. *Journal of the Franklin Institute* (August 1952).

A. M. Gaudin: *Principles of Mineral Dressing.* McGraw-Hill Book Co., Inc., New York, 1939.

Mather Mine Uses Pipeline Concrete in Underground Operations

by Harry C. Swanson

Fig. 1—Pneumatic placer underground. Note telephone for crew placing concrete.

TRANSPORTING concrete from mixer to forms has always been a problem. Twenty-five years ago this task was generally accomplished by means of wheelbarrow or concrete buggy. On large dam jobs, as the number of these projects increased, the gantry crane or highline came into use. Today several methods of handling concrete are employed on smaller surface construction jobs, for example, transit-mix trucks or dumpcrete trucks, which have crawler cranes with buckets for placing concrete into forms.

In 1944, during early stages of developing Mather mine *A* shaft, several large underground concrete jobs were necessary. At this time the Cleveland-Cliffs Iron Co. purchased the first pumpcrete machine, introduced by the Chain Belt Co. of Milwaukee. The machine was used to pour approximately 200 cu yd of concrete for a dam, or bulkhead, located 400 ft from the shaft. Concrete was mixed on surface, lowered down the shaft 1000 ft in a 2-cu yd bucket hung under one skip, spouted into the bowl of the pumpcrete machine from the bucket, and pumped directly into the forms.

Since the day of the first pipeline concrete in 1944 to the present time, other equipment and other methods have been developed to permit transportation of concrete by pipeline through vertical and horizontal distances totaling 1 mile from mixer to forms. Much of the efficiency in present handling of underground concrete can be credited to the Bethlehem Cornwall mines, where concrete was transported through 6-in. pipe for great distances down an inclined shaft and along levels into forms.[1]

During initial development of Mather mine *B* shaft, with concrete work under way on two or more levels at one time, the pneumatic concrete placer, Fig. 1, was selected as best adapted for underground concrete transportation. The ¾-cu yd pneumatic placer is a small machine readily moved from one location in the mine to another. It can be equipped with two sets of mine car wheels, which will permit moving on regular mine tracks. It is therefore possible to send concrete through the pipe at great velocity; the pipeline is clean after each shot except for the film of cement adhering to the inside. With the proper slump in the concrete, it is possible to shoot concrete 2000 ft with this machine, using the mine supply of compressed air at 95 psi.

This equipment was first used at Mather mine *B* shaft to concrete slusher drifts, Figs. 2 and 3, and finger raises located about 2000 ft from the shaft. In several instances there were bends into crosscuts and up vertical distances into the forms.

For the first pours two placers were used. The first was located near the shaft where the concrete could be spouted into it from a 2-cu yd concrete bucket on the cage. The second was set on the side of the drift at a point approximately 1500 ft from the shaft. The concrete was shot directly into the second placer from the first unit and from the second machine directly into the forms. After completion of several pours with the two machines, a trial pour with only one placer located at the shaft proved that the second placer could be eliminated. Since then all pours have been successfully completed with only one placer underground.

As production of iron ore from the mine increased and the development program expanded, use of the cage for handling mine supplies and concrete became a major problem. This brought about the first attempt at shooting concrete vertically down the shaft for 2600 ft. A 6-in. pipeline with victaulic couplings installed during shaft sinking was used for the trial. One placer was set on surface 250 ft from the collar of the shaft so concrete could be spouted directly into it from the mixer. This machine shot the concrete 250 ft horizontally on surface to the shaft, 2600 ft vertically down the shaft, and 100 ft horizontally into the second placer located near the rib of the shaft station or plat. The second machine shot the batch into the forms, about 2000 ft. Total distance horizontally and vertically was 4800 ft. The entire time cycle for a ¾-cu yd batch of concrete from the mixer on surface to the forms underground totaled about 5 min. During the past two years the two-placer method from the mixer on surface to the forms underground has proved a very efficient means of transporting underground concrete. Advantages of using pipeline concrete are as follows:

1—Interference with normal mining operation is eliminated. When the cage, skips, mine cars, or mine openings are used for transporting concrete and materials used for making concrete, mine operation suffers in one way or another.

H. C. SWANSON is District Superintendent, Open Pit Mines, Cleveland-Cliffs Iron Co., Ishpeming, Mich.

Discussion on this paper, TP 2765A, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, Nov. 20, 1953. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 320. The intervening non-Transactions pages appeared in Mining Engineering.

Fig. 2—View of drift after concreting during development of undercut for block cave.

Fig. 3—Concreted drift after approximately 100,000 tons of ore were drawn.

Fig. 4—Form used for concreting slusher drifts.

2—When concrete can be delivered underground at reasonable cost, it can be used to replace more expensive materials. Mine timber, one of the mining supplies that have increased considerably in cost in the past few years, can be eliminated and replaced by concrete for ground support. The concrete-lined drift or raise will outlast several replacements of timber, which is also expensive to install. For block-caving slusher drifts, Fig. 2, where substantial ground support is required, concrete serves well. A screw-jack type of traveling form, Fig. 4, can be used for this work, reducing form costs to a minimum. The only carpenter work required is installation of the bulkhead at the end of the form.

3—When concrete is used for ground support, the miner has neater, safer working conditions. The ever-present fire hazard is also eliminated.

Recent installation of batching plants and bulk cement handling equipment, Figs. 5 and 6, at the Mather shafts and the Negaunee mine has further reduced the cost of concrete. All labor is eliminated, except for hooking the screw conveyor to the hopper car. Previously a sack of cement was handled four times before it reached the concrete mixer. Allowing for truck time and labor, this amounted to 15¢ or 20¢ per bag of cement, approximately $1.00 per cu yd of concrete. Further reduction in cost has resulted from installation of a bucket elevator to fill the aggregate bins. Washed sand and gravel delivered to the mine plant by truck from a local supplier is dumped directly into a hopper and raised into the 300-ton bins by the bucket elevator.

When a placer is used the shorter the bend in a pipeline the less wear in the pipe. In the construction of a right-angle bend a blanked-off tee did the best job. It was found when the plug on the tee was removed after a pour that the blanked-off end of the tee was always free of concrete. The only explanation for this is that the air space acts as a cushion.

To prevent excessive wear a pipeline used with pneumatic placer must be installed as straight as possible between right angles. A slight gradual bend will result in worn-out pipe after a small amount of concrete passes through. Vertical pipe installed off plumb wears excessively. Pipe must be securely fastened to prevent side movement while the shot is being made, as vibration or slapping back and forth will also cause the pipe to wear. Horizontal pipe should be installed as true to line and grade as possible. To make compound or reverse bends such as those required for connections from crosscuts to slusher drifts, a heavy duty 6-in. hose with nipples grooved for victaulic couplings on each end has proved most satisfactory. After several hundred yards of concrete have been poured, victaulic couplings along a horizontal run of pipe should be loosened, two at a time, and the pipe given a quarter turn. This procedure will even out the wear around the perimeter of the pipe, especially in difficult spots. A clean cut through a section of worn-out pipe removed from the troublesome section of the shaft which had not been turned showed that all the original thickness of the pipe remained for three-fourths of the distance around the pipe.

This paper would not be complete without a word of caution regarding a duty that is hazardous if not properly performed. Occasional plugs will occur, even if there are good operators and the proper mix is used, although lost time from these troubles decreases as operators gain experience. The first step

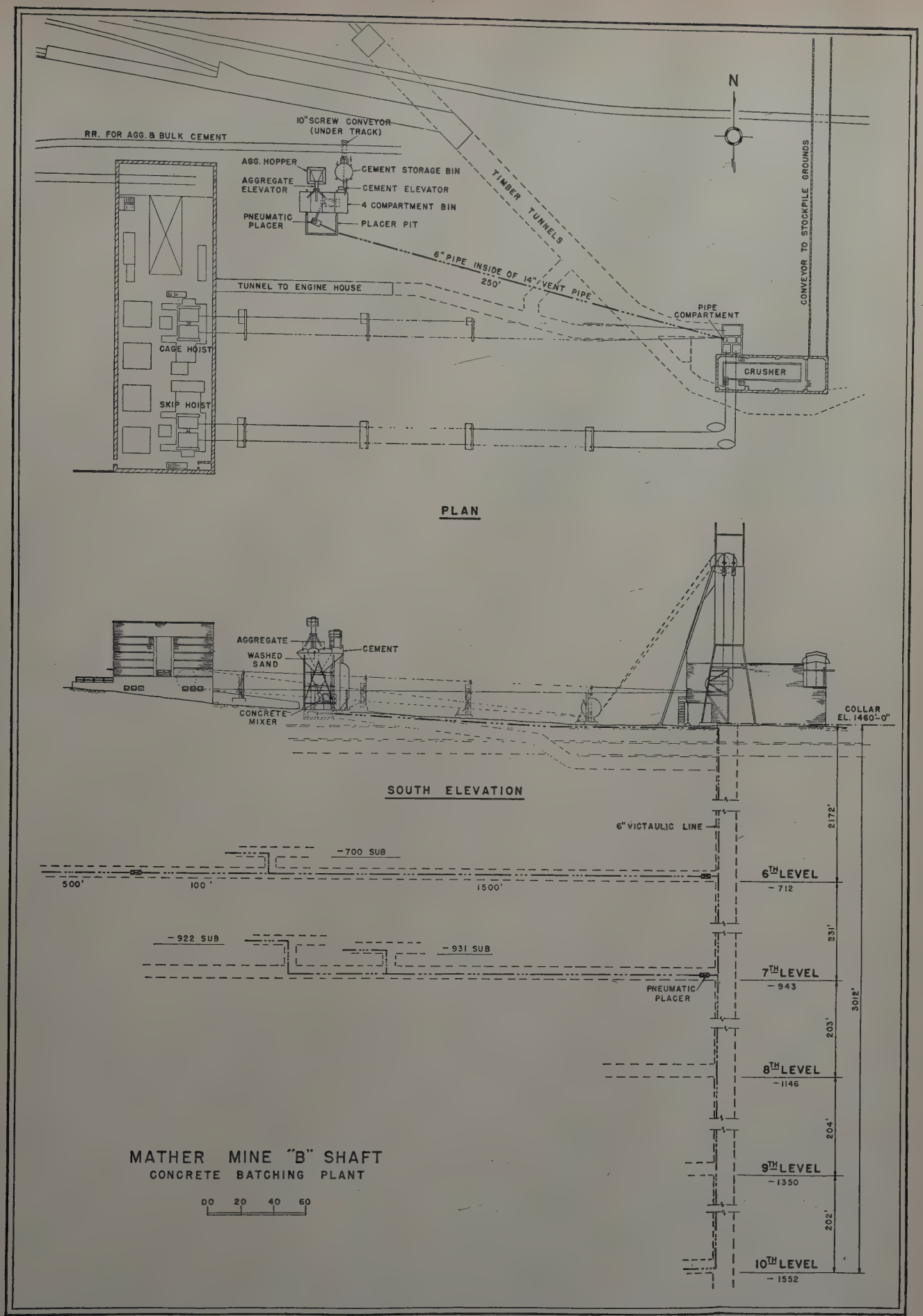


Fig. 5—Plan and section showing route of concrete from batching plant on surface to drifts on 6th and 7th levels.

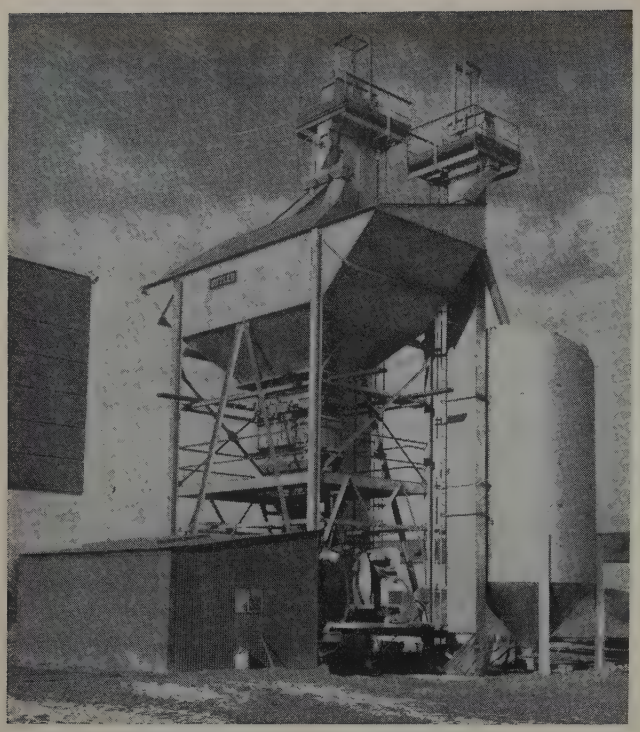


Fig. 6—The new batching plant with bulk cement storage silo.

in relieving a pipe of plug is to tap the plugged section with a hammer, using air pressure on the placer. If this does not set the plug moving, the next step is to bleed the placer of air pressure. The pipeline should then be broken at a short distance beyond the plugged area, air pressure again applied to the line, and tapping of the plugged section resumed with all workers standing clear of the opening in the pipe. At times it becomes necessary to break the pipe through the plugged section, and here again, care should be taken to have the placer free of air pressure. Occasionally two plugs will trap compressed air between them. This is the most dangerous condition. Tapping the pipe before attempting to relieve a plug will indicate which sections are filled with concrete and which sections may have trapped air. This can be detected by the difference in the ring from the tap of the pipe. All men who are working on a plugged pipe should wear safety goggles.

A separate telephone system not connected with any other mine operation is installed for use of the crew placing concrete. A telephone is installed at each of the two placers and one at the form end of the pipe. No concrete is shot until the men at the second placer and the form have been telephoned. Installation of this system has also proved valuable in expediting other materials to the concrete crew.

Wetting Agent in Concrete Used to Lubricate Pipe

Cement and water act as lubricants in the pipe when the pneumatic placed is used. Before any concrete is shot, one placer full of water is shot through to wet the inside perimeter. The first batch of concrete should be mixed richer in cement than the following batches, as a portion of cement in the first batch will adhere to the inside of the full length of the pipe. Additional cement required in the first batch varies with length of pipe; for long shots as much as two bags may be added. After each pour one batch of water should be shot through to clean the pipelines, and if necessary two batches.

Reference

[1] Joseph Bernhardt: Use of Concrete Underground. *Trans.* AIME (1950) **187**, pp. 355-358.

Effects of Oxidation of Coals on Their Flotation Properties

by Shiou-Chuan Sun

THE effects of oxidation on the flotation behavior of sulphide minerals have been extensively studied,[1-3] but no similar study has been made of coals. Coals of bituminous and lower rank undergo atmospheric oxidation during mining and storage, and their degree of oxidizing increases with temperature[4, 5] and time of exposure.[6] The problem of recovering fine oxidized coal from culm piles, rivers, and streams by froth flotation has become increasingly important to the coal industry as well as to those concerned with air and stream pollution. This paper describes the flotation behavior of oxidized and unoxidized coals and carbons in the presence of oily collectors and presents the reagents found effective in the flotation of oxidized coals and carbons.

Except where otherwise stated, flotation tests were performed in a 50-g Denver flotation cell. Solid feed for each test was 30 g, and the pulp dilution was kept at 8.5. The −65 mesh coal sample was wetted by agitation with water in the flotation cell for 6 min, then conditioned with a predetermined amount of collector for 2 min, and finally floated with pine oil as frother for 3 min.

The oxidation of coals and carbons was performed in a vertical, electrically heated glass tube, shown in Fig. 1. One hundred grams of −65 mesh coal were fed into the glass tube and heated to 10° to 20°C below the predetermined temperature of oxidation. Compressed air was admitted to the tube at a rate of 0.05 cu ft per min and the temperature was controlled by a variable voltage regulator. The oxi-

S. C. SUN, Member AIME, is Associate Professor, Department of Mineral Engineering, The Pennsylvania State University, State College, Pa.

Discussion on this paper, TP 3681F, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, April 22, 1953. St. Louis Meeting, February 1951.

dation was continued until the equivalent weight[7] of the oxidized product no longer decreased. Generally, the oxidation for all coals was ended at about 250 hr, except that for subbituminous and lignitic coals which was ended at approximately 150 hr.

Coals and carbonaceous materials were analyzed for carbon and hydrogen in accordance with the standard ASTM procedures. The difference between the sum of the percentages of carbon and hydrogen and 100 pct was considered as oxygen. The methods used for the determination of the equivalent weights and the solubilities in 1 N sodium hydroxide of the oxidized coals can be found in the literature.[7, 8]

Flotation tests of unoxidized and oxidized coals and carbons were performed with purified petroleum light oil as collector. Results are given in Fig. 2. Similar results, not presented, were also obtained by parallel tests with other oily collectors such as kerosene, decane, dodecane, and tetradecane. Fig. 2 shows that floatabilities of the tested coals in the presence of an oily collector are: 1—decreased generally in the order of bituminous, subbituminous, anthracite, and lignite; 2—lowered gradually by the increase of the extent of oxidation; and 3—little affected and occasionally benefited by the superficial oxidation at low temperatures.

Upon review of the analytical data of Table I, the above flotation phenomena can be explained by the *surface components* hypothesis.[9] For example, considering point 1, the high floatability of bituminous coal is attributed to its high contents of floatable components including hydrogen and carbon, in conjunction with its relatively low percentages of nonfloatable components including water, oxygen, and ash. The comparatively low floatability of subbituminous coal is caused by a relatively high content of water and oxygen as well as a slight decrease in hydrogen and carbon contents. The low percentage of hydrogen is considered the main reason for the still lower floatability of anthracite coal, whereas the extremely high percentages of water and oxygen cause the very low floatability of lignite. Regarding point 2, the low floatability of sufficiently oxidized coals and carbons is caused by the notable increase of their oxygen and ash contents, in combination with the sharp reduction of their hydrogen and carbon contents. In the process of oxidation, the floatable components of coals were oxidized not only to liberate gases but also to produce oxidation products. Thus the original oil-avid surface of the coal was

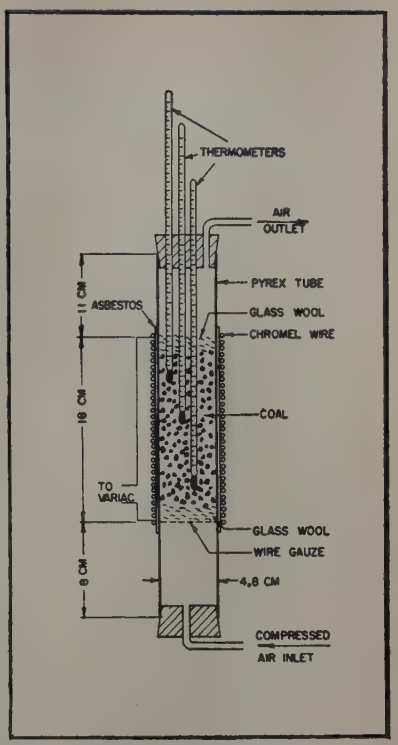


Fig. 1—An electrically heated apparatus for controlled air-oxidation.

Table I. The Chemical Compositions and Sources of the Test Coals and Carbons*

Coal or Carbon	Oxidation Temperature, °C	Oxidation Time, Hr	Chemical Analysis, Pct Received Basis Ash	Received Basis H_2O	Dry and Ash-Free Basis C	Dry and Ash-Free Basis H	Dry and Ash-Free Basis O	Equivalent Weight, Dry and Ash-Free Basis	Solubility in 1N Alkali, Pct Dry and Ash-Free Basis	pH of Pulp Containing 30 G Solid and 250 Cc Tap Water
Lignite, Velva, N. Dak.	Unoxid.		8.6	17.5	75.0	3.99	19.37			
	150	175	9.6	2.4	67.2	3.6	29.2	216	97	
	200	175	13.9	1.6	65.9	2.5	31.6	185	102	
Subbituminous, Monarch, Wyo.	Unoxid.		6.0	14.2	78.0	4.4	17.6			
	150	127	6.5	2.9	66.8	3.3	29.9	196	90	
	200	140	8.3	4.3	66.7	2.6	30.7	198	98	
	250	140	13.4	2.0	66.3	2.1	31.6	190	102	
High volatile, A Pittsburgh, Pa.	Unoxid.		9.5	4.6	87.5	5.5	7.0			
	150	245	8.3	3.9	74.6	3.7	21.7	276	78	
	200	245	10.0	4.9	69.7	2.3	28.0	185	93	
	250	245	8.9	5.6	67.4	2.2	30.4	164	97	
	300	245	25.6	1.3	65.1	2.0	32.9	165	99	
Medium volatile No. 1 Upper Freeport, Pennsylvania	Unoxid.		7.7	0.4	86.9	5.3	7.8			6.8
	150	230	7.8	2.9	75.6	3.7	20.7	366	12	
	200	190	8.0	0.7	71.7	2.6	25.7	198	96	3.3
	250	235	15.9	0.5	71.7	2.0	26.3	176	88	3.1
	300	235	38.8	0.7	71.1	2.1	26.8	171	88	
Medium volatile No. 2 Sewell, W. Va.	Unoxid.		3.1	1.1	81.5	5.1	13.4			6.7
	150	240	2.9	2.3	80.0	3.7	16.3	451	5	5.1
	200	210	3.0	1.4	71.7	2.3	26.0	203	90	4.3
	250	141	3.4	0.7	71.3	2.1	26.6	188	93	4.0
	300	252	13.0	2.7	66.2	1.9	31.9	184	99	
Low volatile, Pocahontas No. 3, W. Va.	Unoxid.		2.9	0.6	90.5	4.6	4.9			6.5
	150	237	3.5	0.6	84.3	3.8	11.9	1245	7	5.5
	200	240	2.9	0.8	75.0	2.6	22.4	236	91	4.5
	250	262	3.5	0.9	70.9	2.0	27.1	194	99	4.1
	300	389	6.5	1.4	68.6	1.8	29.6	182	91	
Anthracite, Southern field, Penna.	Unoxid.		9.8	1.8	95.6	1.7	2.7			
	150	218	10.8	1.9	94.6	1.8	3.6	>2000	7	
	200	240	10.6	2.3	93.7	1.9	4.4	>2500	6	
	250	230	9.1	3.3	93.2	1.8	5.0	>1800	8	
	300	232	12.0	2.4	85.3	1.5	13.2	737	6	
	350	303	29.0	3.7	74.4	1.8	23.8	239	12	
Graphite, spectroscopic grade, National Carbon Co.			0.2	0.1	99.5	0.2	0.3			6.8
Lamp black, Martin, Spear Carbon Co.			0.2	1.1	95.5	0.6	3.9			6.9

* Analytical work was performed by Dr. L. D. Friedman and Professor C. R. Kinney.

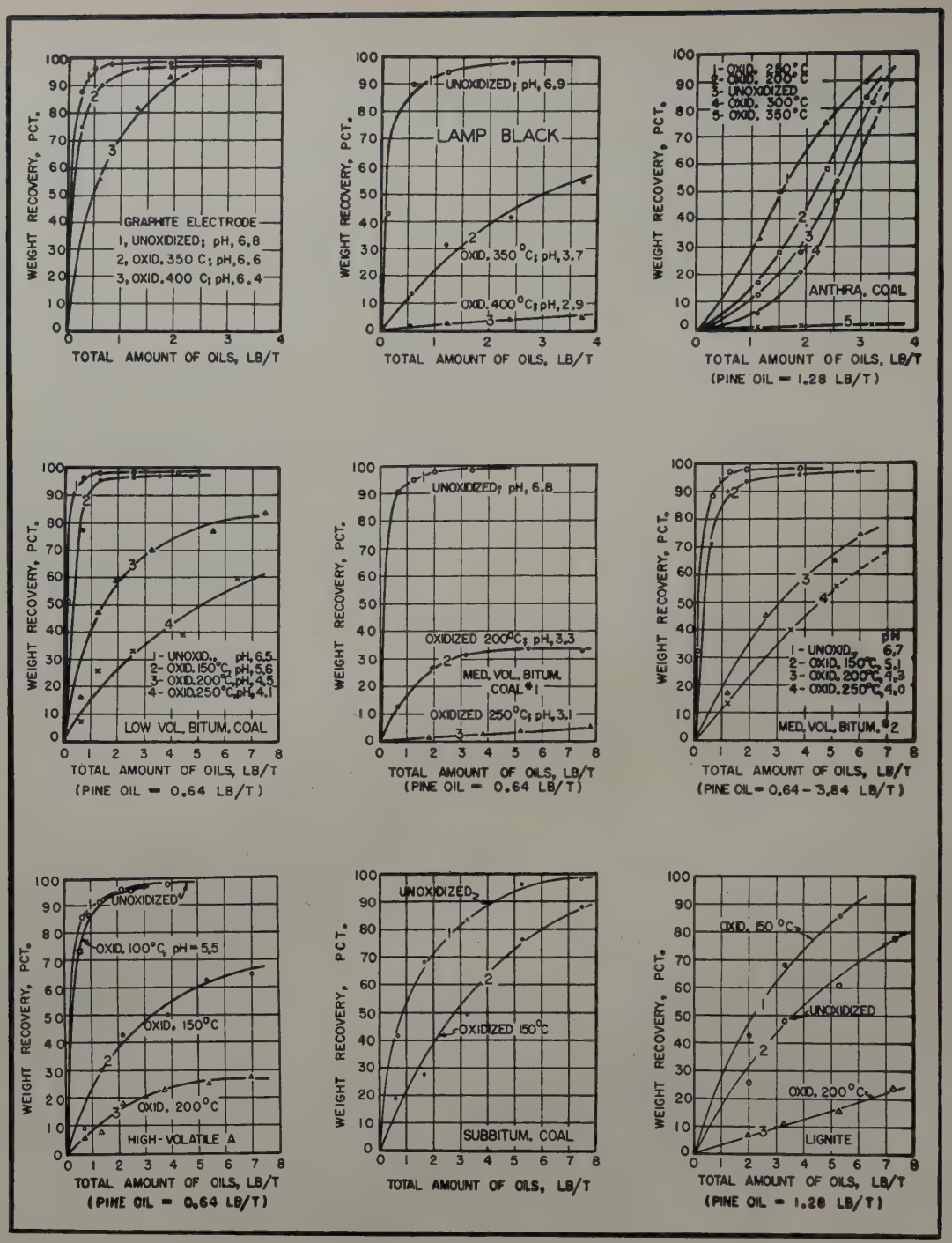


Fig. 2—Floatability of unoxidized and oxidized coals and carbons with purified petroleum light oil as collector.

oxygenized to become water-avid and oil-repellent, owing to the increase of oxygen-containing compounds and ash. Considering point 3, the fact that lignite and anthracite gained some floatability through superficial oxidation at low temperatures is attributed to the lack of pernicious deterioration of their surfaces and to the collecting and/or frothing properties of their water-soluble oxidation products. Table I shows that the oxidation of the lignite at 150°C resulted in a sharp decrease of its moisture content and a relatively small reduction of its hydrogen and carbon contents. The benefit from the dehydration is more than sufficient to compensate the harm by the slight decrease of hydrogen and carbon contents. Table I shows also that the slightly oxidized anthracite exhibited no substantial change in chemical composition. From the work of Bone[10] and Kinney,[11] it is speculated that the oxidation products resulting from the superficial oxidation of anthracite are chiefly benzenoid acids with large non-polar groups and high molecular weights. These acids are weak frothers and some of them are weak collectors, as can be visualized from the tested data of Fig. 3 and Table III.

The chemical properties of the oxidation products of coals have been studied by many investigators. It is generally accepted that the oxidation of coals can be divided into three stages.[4] Stage 1 is a superficial oxidation characterized by the formation of coal-oxygen complexes[5, 12, 13] with acidic properties. In stage 2, a large portion of the organic components of coal, including hydrocarbons and carbon, is transformed into hydroxycarboxylic acids, generally called humic acids.[8, 14] A small amount of ammonia

and nitrate ion[15] may also be liberated from the nitrogen-containing carbon compounds. The carboxyl groups of the humic acids, leading to average equivalent weights[7] of 200 to 300, are responsible for the oxidized coal being alkali-soluble and acid-insoluble. In stage 3, the humic acids are further degradated to water-soluble acids.[11, 16, 17] Under the same oxidation conditions, the ratio between the benzoid and oxalic acids in the oxidation products of coals[10, 11] increases with the maturity of the coal. The resistivity of coals to oxidation increases with their ranks, whereas the resistivity of petrographic components[18, 19] to oxidation increases in the order of vitrain, clarain, durain, and fusain.

Nonfloatability of the Water-Insoluble Oxidation Products of Coal Surfaces

An attempt was made to determine whether the low floatability of a mildly oxidized coal is caused by its water-insoluble oxidized surface or its water-soluble oxidation products in the flotation pulp. The test results, as given in Table II, show that the water-insoluble surface of the oxidized coal is chiefly blamed for its low floatability. This is due to the fact that the surfaces of considerably oxidized coals, predominated by hydroxyl and carboxyl groups,[4-7] are water-avid and oil-repellent. A comparison between tests 2 and 3 of Table II shows that

Table II. Flotation of Pittsburgh Bituminous Coal Under Various Conditions with 1.02 Lb per Ton Purified Kerosene and 0.64 Lb per Ton Pine Oil

Test No.	Oxidation	Coal Treated Before Flotation	pH of Pulp, 30 G Coal and 125 CC Water	pH of Filtrate of Tailing	Recovery, Wt Pct
1	Unoxid.	None	6.9	6.8	100.0
2	200°C	None	3.9	4.0	37.5
3	200°C	Washed 10 times with distilled water.	5.0	4.9	30.4
4	200°C	First conditioned in a warm NaOH solution of 1.0 N concentration for 10 min, then washed with distilled water 32 times	7.2	7.0	36.2

the floatability of the oxidized coal is slightly benefited or at least is not appreciably depressed by the presence of its water-soluble oxidation products in the flotation pulp. Tests 3 and 4 of the same table show that once the original surfaces of coals are appreciably deteriorated by oxidation, they cannot be restored through washing with water or alkaline solutions.

Effect of Water-Soluble Oxidation Products On The Floatability of Unoxidized Coal

The influence of water-soluble oxidation products and some of their constituents on the floatability of Pittsburgh bituminous coal was ascertained by means of flotation, quantitative bubble pick-up,[20] and frothing tests.[21] The experimental data, as given in Fig. 3 and Table III, indicate that, in the flotation of unoxidized coals, the behavior of water-soluble products resulting from various stages of oxidation of coals is generally as follows: A—stage 1, weak collectors; B—stage 2, mixtures of weak collectors and weak depressants; and C—stage 3, mixtures of weak and moderate depressants. The same data indicate also that the water-soluble products resulting from all the stages of oxidation of coals are weak frothers. As a whole, the effect of the water-soluble products in lowering floatability is relatively unimportant, as compared with the important role played by the water-insoluble oxygenated surfaces of coals.

Fig. 3A and tests 1 to 4 of Table III show that the tested benzene carboxylic acids, which may be produced in stage 1 oxidation of coals,[10] are capable of increasing the flotation recovery of the coal, because they are weak collectors as well as weak frothers. The same data show also that the collecting and frothing powers of these acids generally decrease with an increase of their carboxyl groups.

It is deduced from the previous work on humic acids[11, 16, 17] that the water-soluble products of stage 2 oxidation consist chiefly of aromatic hydroxycarboxylic acids and only partly of short-chain alphatic carboxylic acids as well as alphatic hydroxycarboxylic acids. Fig. 3C and tests 5 to 10 of Table III show that the tested aromatic hydroxycarboxylic acids are either weak depressants or weak collectors, depending upon whether their molecules are attached with sufficient number of polar groups,

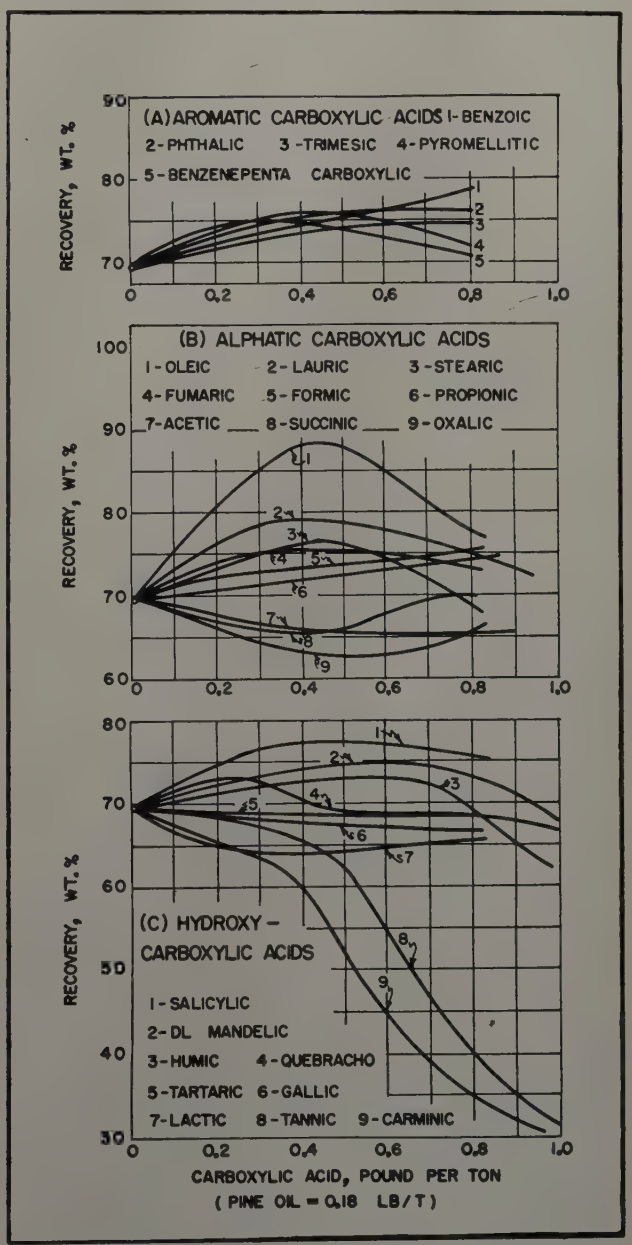


Fig. 3—Effect of water-soluble oxidation products on the flotation of Pittsburgh bituminous coal with 0.18 lb per ton of pine oil.

especially hydroxyl groups. For example, salicylic acid with one hydroxyl group is a weak collector, whereas gallic acid with three hydroxyl groups is a weak depressant. A considerable depression was achieved by both tannic and carminic acids, because of the large number of their polar groups as well as the massive size of their molecules. Nevertheless, these two acids have never been identified in the oxidation products of coals. In a similar way, Fig. 3B and tests 11 to 19 of Table III show that the tested long-chain alphatic carboxylic acids, which are not expected to be found in the oxidation products of coals, are moderate collectors. A short-chain alphatic carboxylic acid can depress coal slightly only when the ratio between the number of carbon atoms of its nonpolar group and the number of its carboxyl group is not more than one. This can be exemplified by the weak depression contributed by acetic acid,[11] succinic acid, and oxalic acid.[17] Exceptions to this rule are formic and fumaric acids. The fact that lactic and tartaric acids behaved as weak depressants is chiefly caused by their hydroxyl groups. Generally, the water-soluble products of stage 2 oxidation are mixtures of weak collectors and weak depressants, resulting in no appreciable effect on the floatability of coals. This is particularly true when the concentration of these water-soluble products in the coal pulp is low.

In stage 3 oxidation, the oxidation products of stage 2 are further degradated not only to increase the content of alphatic hydroxycarboxylic acids, but also to increase the number of hydroxyl and carboxyl groups of the aromatic hydroxycarboxylic acids. As a result, the water-soluble products of stage 3 are expected to be mixtures of weak and moderate depressants.

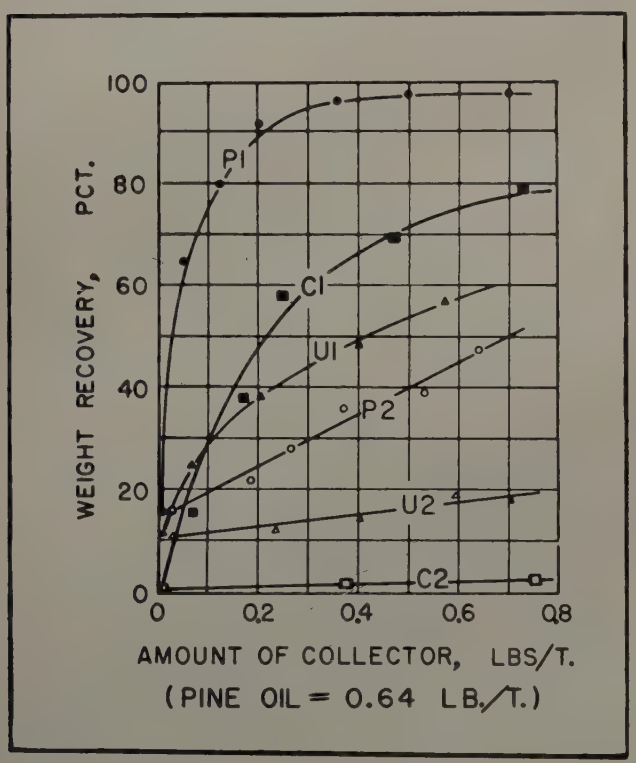


Fig. 4—Flotation of oxidized bituminous coals and coke with cationic reagents and oils. Curve P_1 and P_2: Pocahontas bituminous coal, oxidized at 200°C and floated with lauryl amine and petroleum light oil respectively. Curves C_1 and C_2: High-temperature coke, Koppers Coke Co., Philadelphia, Pa., oxidized with concentrated nitric acid and floated with lauryl amine hydrochloride and kerosene respectively. Curves U_1 and U_2: Upper Freeport bituminous coal, oxidized at 200°C and floated with rosin amine-D acetate and petroleum light oil respectively.

Table III. The Results of Flotation, Bubble Pick-Up, and Frothing Tests of Organic Acids Which May Occur in the Water-Soluble Oxidation Products of Coals

Test No.	Organic Acid	Solubility,* G Per Liter	Net Flotation Recovery of Pittsburgh Bituminous Coal, Pct	Bubble Pick-up Coefficient of 48x65 Mesh Pittsburgh Bituminous Coal, Pct	Frothability Index of Organic Acid, Using N-Hexyl Alcohol as Standard
1	Benzoic	1.8	6.8	20	50.4
2	Phthalic	5.4	6.7	7	46.1
3	Trimesic	26.9	4.5	2	—
4	Pyromellitic	14.2	5.0	1	23.2
5	Humic	0.1**	3.1	0	30.8
6	Salicylic	1.8	7.5	18	—
7	Dl mandelic	160.0	4.9	16	—
8	Gallic	11.6	−3.2	−2	23.2
9	Tannic	s	−15.0	−12	—
10	Carminic	v.s.	−25.0	−15	15.4
11	Oleic	i.	15.0	16	—
12	Lauric	i.	7.6	14	23.2
13	Stearic	0.34	4.4	12	—
14	Fumaric	7	5.0	1	23.2
15	Propionic	s.	2.8	0	69.1
16	Acetic	s.	−4.9	−2	—
17	Oxalic	95.0	−7.5	−3	23.2
18	Tartaric	206.0	−2.0	−2	23.2
19	Lactic	s.	−5.1	−6	61.6

* i, insoluble; s, soluble; v.s., very soluble.
** Approximation.

Referring to Table III, the term *net flotation* recovery denotes the difference between the flotation weight per cent recovery of Pittsburgh bituminous coal with 0.6 lb per ton organic acid and 0.18 lb per ton pine oil and that of the same coal with 0.18 lb per ton pine oil alone at the same pH. The term *bubble pick-up coefficient* represents the difference between the bubble pick-up index[20] of 48x65-mesh Pittsburgh bituminous coal in a solution of 50 mg per liter organic acid and that of the same coal in distilled water only, at the same pH. The frothability index[21] of organic acids was tested at 19.26 mg per liter concentration, 7.0 ± 0.2 pH, and 71.6 cc per sq cm per min rate of aeration, using n-hexyl alcohol as standard.

Flotation of Oxidized Coals and Cokes With Cationic Reagents

Tests were performed to find suitable collectors for the flotation of considerably oxidized coals and carbons. The experimental results, as given in Fig. 4, show that coals and carbon oxidized with air as well as with concentrated nitric acid[15] can be effectively floated by certain cationic reagents but not by the currently used oily collectors. This may be caused by the fact that chemical reaction and ionic attraction can take place between the positively charged polar groups[22] of amines and the negatively charged ions, particularly carboxyl groups, of the oxidized surfaces of coals and carbons. The speculation that the oxidized surfaces of coals and carbons are dominated by anions was experimentally verified by testing the reversibility of coals with an electrostatic separator. Tests were also made on floating oxidized coals with mixtures of iso-amyl amine and kerosene. The results, not presented, showed that the flotation recoveries generally decreased with an increase of the percentages of kerosene in the blend. The same thing was also true for mixtures of lauryl amine and petroleum light oil.

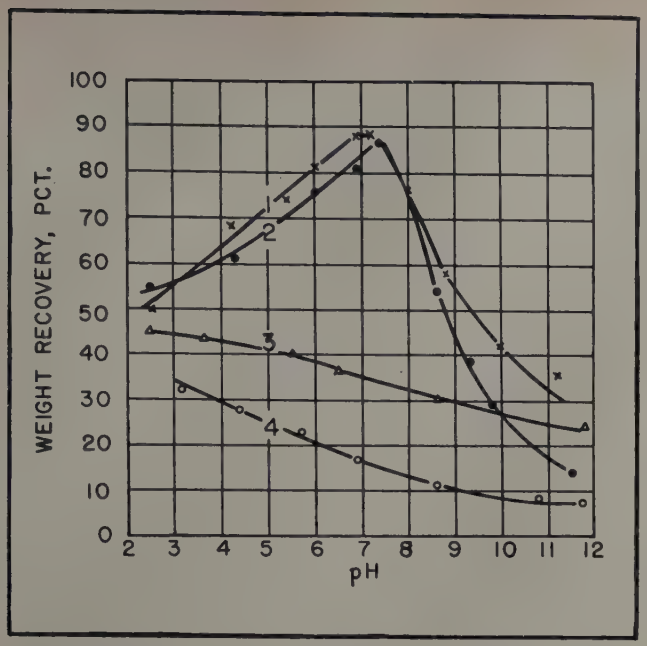


Fig. 5—Effect of pH on the flotation of unoxidized and oxidized coals. Curve 1: Philadelphia and Reading anthracite coal, floated with 1.95 lb per ton Squibb mineral oil and 0.6 lb per ton pine oil. Curve 2: Bituminous coal, Snowshoe, Pa., floated with 0.133 lb per ton borneol. Curve 3: Bituminous coal, Pittsburgh, Pa., oxidized at 200°C for 200 hr and floated with 0.7 lb per ton L-Am-HCl and 0.19 lb per ton pine oil. Curve 4: Bituminous coal, upper Freeport, Pa., oxidized at 200°C and floated with 8.8 lb per ton petroleum light oil, 1.28 lb per ton pine oil.

The effect of pH on the flotation of unoxidized and oxidized coals with various collectors was tested, and the results are given in Fig. 5. Referring to this figure, Curves 1 and 2 show that the optimum pH values for unoxidized anthracite and bituminous coals in presence of oily collectors are respectively 7.0 and 7.5. Curves 3 and 4 show that with lauryl amine hydrochloride and petroleum light oil as collectors the floatabilities of oxidized coals generally increase with the decrease of pH value.

Summary

1—The floatabilities of coals and carbons in the presence of a neutral oily collector generally decrease with an increase of the extent of their oxidation.

2—With neutral oils as collectors, the floatabilities of coals, particularly anthracite and lignite, are little affected and occasionally benefited by the superficial oxidation at low temperatures.

3—The low floatability of a mildly oxidized coal is caused chiefly by the water-insoluble oxidation products of its oxidized surface.

4—Generally the flotation of unoxidized coals with oily collectors is very little affected by the presence of a small amount of water-soluble oxidation products of coals.

5—Certain cationic reagents are found to be effective in the flotation of oxidized coals and cokes.

6—The floatabilities of oxidized coals in presence of oily collectors as well as cationic collectors generally increase with the decrease of pH value.

Acknowledgment

This writer is indebted to L. D. Friedman and C. R. Kinney for providing some of the coal samples and the chemical analyses. He also wishes to thank H. L. Kee for his participation in the oxidation of coals, G. Simkovich for proof-reading the manuscript, and D. R. Mitchell and H. B. Charmbury for their encouragement.

References

[1] A. M. Gaudin. H. Glover, M. S. Hansen, and C. W. Orr: Flotation Fundamentals, Tech. paper 1, Utah Engr. Expt. Station, (1928).

[2] E. C. Plante and K. L. Sutherland: Effects of Oxidation of Sulphide Minerals on Their Flotation Properties. *Trans.* AIME (1949) **183**, p. 160.

[3] E. C. Plante: Flotation of Unoxidized and Oxidized Minerals: Antimonite, Arsenopyrite, Covellite, Lollingite, Marcasite, Orpiment, Pyrrhotite, and Tetrahedrite. *Trans.* AIME (1949) **183**, p. 189.

[4] H. H. Lowry: The Chemistry of Coal Utilization. **1**, p. 346. John Wiley and Sons, Inc., New York, 1945.

[5] L. D. Schmidt, J. L. Elder, and J. D. Davis: Atmospheric Oxidation of Coal at Moderate Temperatures. *Industrial and Engineering Chemistry* (1940) **32**, p. 548.

[6] G. R. Yohe and M. H. Wilt: The Oxidizing Power of Illinois Coal. 2. The Effect of Extended Time. Circ. No. 84, Illinois State Geological Survey, Urbana, Ill. (1942).

[7] C. F. Brown and A. R. Collett: A Potentiometric Study of the Humic Acids Derived From Coal. *Fuel* (December 1938) **17**, p. 350.

[8] L. D. Friedman and C. R. Kinney: Humic Acids From Coal. *Industrial and Engineering Chemistry* (December 1950) **42**, p. 2525.

[9] S. C. Sun: Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals. *Trans.* AIME (1954) **199**, p. 67.

[10] W. A. Bone and G. W. Himus: *Coal, Its Constitution and Uses,* pp. 186-193. Longmans, Green and Co., New York (1936).

[11] C. R. Kinney: The Source of Acetic Acid Obtained by Oxidation of Coal. *Journal of the American Chemical Society* (1947) **69**, p. 284.

[12] A. B. Lamb and L. W. Elder, Jr.: The Electromotive Activation of Oxygen. *Journal of the American Chemical Society* (1931) **53**, p. 157.

[13] G. R. Yohe and C. A. Harman: The Oxidizing Power of Illinois Coal. 1. The Reaction with Titanous Chloride, Circ. No. 70, Illinois State Geological Survey, Urbana, Ill. (1941).

[14] W. Fuchs and A. G. Sandhoff: Investigations Concerning Hydroxy Carboxylic Acids from Bituminous Coal. *Fuel* (1940) **19**, p. 45.

[15] H. B. Charmbury, J. W. Eckerd, J. S. LaTorre, and C. R. Kinney: The Chemistry of Nitrogen in Humic Acids From Nitric Acid-Treated Coal. *Journal of the American Chemical Society* (1945) **67**, p. 625.

[16] B. Juettner, R. C. Smith, and H. C. Howard: Oxidation of a Pittsburgh Seam Bituminous Coal and Low Temperature Coke by Alkaline Permanganate. *Journal of the American Chemical Society* (1937) **59**, p. 236.

[17] G. T. Morgan and J. I. Jones: Studies on the Constitution of Coal, Part 1. Progressive Oxidation of Coal Humic Acids. *Journal of the Society of Chemical Industry.* London (September 1938) **57**, p. 289.

[18] G. R. Yohe and C. A. Harman: Preparation of Humic Acids From Illinois Coals. *Trans.* Illinois State Acad. Sci., (1939) **32**, No. 2, pp. 134-136.

[19] W. Fuchs, A. W. Gauger, C. C. Hsiao, and C. C. Wright: The Chemistry of the Petrographic Constituents of Bituminous Coal, Penna. State College, Min. Ind. Expt. Sta., *Bull. 23,* (1938).

[20] S. C. Sun and R. C. Troxell: Quantitative Bubble Pick-up Methods. *Trans.* AIME (July 1953) p. 715.

[21] S. C. Sun: Frothing Characteristics of Pine Oils in Flotation. *Trans.* AIME (1952) **193**, pp. 65-71.

[22] A. M. Gaudin and S. C. Sun: Correlation Between Mineral Behavior in Cataphoresis and in Flotation. *Trans.* AIME (1946) **169**, p. 347.

New Techniques for Evaluating Natural Corundum Ores

by Howard F. Carl, Howard W. Jaffe, and Arthur Hockman

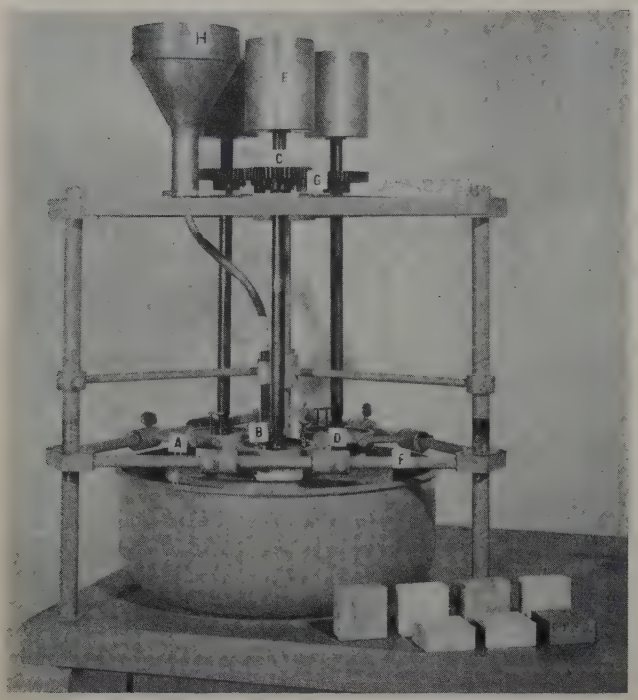


Fig. 1 (left)—National Bureau of Standards laboratory abrasion machine.

THE problem of establishing practical techniques for evaluating natural corundum ores arose from the desire to improve the existing purchase specifications for crystal corundum procured by the Federal Government for stockpiling. Such purchase specifications had been developed for a large number of products, including minerals and ores considered to be critical and strategic because of the country's dependence on foreign sources of supply.

It will be apparent to anyone considering the subject of purchase specifications that the determination of whether or not a submittal meets the specifications is a prime consideration. Consequently satisfactory specifications for minerals should not only incorporate physical, chemical, and mineralogical requirements that are practical but should also establish the means by which both buyer and seller can be assured that materials meet stated requirements.

The Munition Board's Material Purchase Specification for corundum, dated February 1954, covered two grades of crystal corundum intended primarily for abrasive use. It established limits of size and of composition: Grade A, at least 95 pct +3 mesh and 92 pct alumina (Al_2O_3); Grade B, at least 95 pct +7 mesh and 90 pct alumina. It also specified that "of each lot of material, not less than 95 percent shall have a hardness equal to or exceeding nine (9) on Moh's Scale."

These specifications were unsatisfactory for several reasons. They did not allow the purchase of lower grades of corundum being used by industry. Neither did they specify exactly what a corundum ore actually was, except that it should contain so much alumina and have a certain scratch hardness. Nothing was said about the abrasive qualities of the material that was to be purchased primarily for abrasive use.

During the period 1946 to 1948 supplies of the two grades defined in the then current specifications continued to decrease. Early in 1949 industry recommended that the finer sizes of corundum known as "crystal corundum concentrates," ¼-in. to 16-mesh material, be added to the stockpile specifications and that lower grades of crystal corundum also be included.

In the spring of 1949 the National Bureau of Standards and the Bureau of Mines were asked by the Munitions Board to cooperate in developing more practical specifications for grading natural corundum ores. The National Bureau of Standards indicated that it would investigate physical test methods for determining abrasive characteristics of corundum, and the Eastern Experiment Station of the Bureau of Mines at College Park, Md., stated that it would consider the mineralogical aspects of the problem.

Members of these two agencies, in consultation with representatives of the Office of Materials Resources of the Munitions Board and of the Federal Supply Service of the General Services Administration, and with the cooperation of industrial suppliers and consumers of abrasive corundum, developed, in a fairly short time, more practical specifications for six grades of corundum ores.

The new specifications, issued January 1951, were for *corundum, natural crystals,* as by this time high-purity synthetic corundum was available and widely used. Nevertheless, for certain specific applications the natural product was preferred over the synthetic material. It has been extensively used in forming snagging wheels. It breaks into more or less cubical particles, whereas artificial corundum fractures into splintery shapes. These produce irregular cuts in glass, while the more symmetrical shape produces a more uniform ground surface. For this reason natural corundum has been preferred for certain types of lens grinding.

Pure corundum is clear, colorless alpha alumina (Al_2O_3), but it is rarely found in nature in this form. The most important gem varieties include the blue and yellow sapphires and the ruby. Minute impurities of titanic oxide impart a blue color,

H. F. CARL, Physicist, is with Eastern Experiment Station, U. S. Bureau of Mines, College Park, Md.; H. W. JAFFE was formerly Petrographer, Eastern Experiment Station, College Park, Md.; and A. HOCKMAN is Materials Engineer, Division of Building Technology, National Bureau of Standards, Washington, D. C.

Discussion on this paper, TP 3742H, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, Sept. 18, 1953. Los Angeles Meeting, February 1953.

ferric oxide a yellow color, and chromic oxide a red color to otherwise pure corundum. It crystallizes in the hexagonal system, rhombohedral class, having as common forms six-sided prisms, barrel-shaped prisms, and hexagonal bi-pyramids. Ore material has a specific gravity of 3.90 to 4.15 and occurs in a wide variety of dull colors. Intimately associated with the corundum may be varying amounts of other minerals, such as biotite, margarite, ilmenite, magnetite, spinel, rutile, apatite, kyanite, and monazite. Emery is a fine-grained variety of corundum that contains magnetite or hematite.

The South African corundum industry, which supplies most of the material imported into the United States, classifies corundum into four groups: A—*Boulder* is rock containing up to 80 pct corundum, broken into convenient sizes for shipping, with no other treatment. B—*Crystal* is corundum in the form of loose crystals larger than ⅛ in. smallest dimension after removal of adhering impurities. This is the common form of export product. C—*Crystal concentrates* are similar to crystal but smaller than ⅛ in. D—*Grain corundum* is the finished product.

Preparation of Samples: Five corundum samples were obtained from the American Abrasive Co.: three African crystal ores, one African crystal concentrate, and one Brazilian crystal ore. Three samples of ores from Bangalore, India, were supplied by the General Services Administration. Four samples of silicon carbide, two each from the Norton Co. and the Carborundum Co., and one sample of synthetic Al_2O_3 from each of these companies were added to the test samples to be compared.

The ore material, as received, was too large for testing. It was therefore decided to grind each sample to an "abrasive grain size of No. 80." No. 80 grain size is defined as follows. Of a 100-g sample shaken for 5 min on a RoTap testing sieve shaker operated at a speed of 290 rpm and 156 taps per min, the allowable limits are: 1—100 pct must pass the No. 60 sieve. 2—Not more than 15 pct shall be retained on the No. 70 sieve. 3—A minimum of 40 pct shall be retained on the No. 80 sieve. 4—An accumulative minimum of 70 pct shall be retained on the No. 80 and No. 100 sieves. 5—A maximum of 3 pct shall pass the No. 120 sieve.*

* The limits of No. 80 size grain have been changed slightly since these tests were made. Current specifications for grain sizes are described in *S.P.R.* 118-50, U. S. Department of Commerce. All sieve sizes are in the U. S. Standard Sieve Series.

Each sample was ground in a rolls crusher to pass No. 40 sieve. It was then put through a pulverizer several times, with alternate sieving and grinding, until a sufficient quantity was obtained that would comply with the above specifications for No. 80 grain size.

In most cases only about 30 pct of the original sample was recovered in the grinding process. The remaining 70 pct was lost in useless fines and dust.

Although the artificial abrasives obtained from their manufacturers were designated as No. 80 grain size, sieve analyses revealed that all contained either or both excessive fine and coarse grains. Portions of each were resieved to obtain a true No. 80 abrasive.

Hardness Determinations: Preliminary hardness studies were made first with a laboratory abrasion machine, shown in Fig. 1. This machine is generally used to test the abrasive hardness of stone and other hard flooring materials and was thought possibly to be satisfactory for testing abrasives themselves. It was subsequently found unsatisfactory. However, in this particular series of tests it was adapted for the testing of the abrasives. A hard, fine-grained, siliceous rock known as *novaculite* was selected as the standard for testing the various samples of abrasive grains. A cast-iron lap, *A*, of 10-in. diam is driven at 45 rpm by a motor mounted below the catch basin. Three specimen holders, *B*, position the novaculite blocks, 2x2x1 in. Gears, *C*, for revolving the specimens, guide rings, *D*, for holding specimens in position, hoppers, *E*, containing steel shot to make a total load on specimens of 2 kg each, are supported by a framework, *F*, and a supporting plate, *G*. A holder for the test abrasive grain, *H*, allows the latter to flow to the lap by gravity.

The standard stone specimens are held on the revolving cast-iron disk under a constant pressure of 2 kg and made to revolve at half the speed of the grinding disk. The abrasive sample under test is fed to the disk at a constant rate. Hardness of the sample is evaluated by the weight loss of the standard specimen after 5 min grinding.

The second series of hardness determinations was made with the National Bureau of Standards field or portable abrasion tester, shown in Fig. 2. This had also been used to test the hardness of flooring materials but was readily adapted for testing the abrasive grains. The machine consists of a notched-steel wheel, *A*, of 6-in. diam and ½-in. thickness, supported by a framework, *B*, and driven by a sprocket drive, *C*. An abrasive feeder drive belt, *D*, turns a rubber conveyor belt, *E*, for the abrasive contained in the funnel, *F*. A trough, *G*, conducts the abrasive directly between the wheel and a polished piece of plate glass, *H*, 12x12x¼ in. An adjustable weight, *I*, provides for a load of 3340 g, about 7½ lb, on the notched wheel as it revolves, and the abrasive grains are fed at a uniform rate to the cutting rim. These may then be caught in a pan at the bottom.

The tests consist in rotating the wheel 25 revolutions and then measuring with a traveling micrometer the lengths of the segments cut in the material being tested. When length of cut and dimensions of the wheel are known, the segment area, *S*, is readily calculated. The latter is a measure of the hardness of the abrasive. Preliminary investigations indicated that five such tests, each with fresh abrasive, were sufficient to produce the necessary accuracy of the indicated results.

Toughness: This test was also made with the field abrasion tester. A portion of each sample of abrasive is fed to the rim of the wheel while the latter is turned 25 times on the plate glass. This operation is then repeated 10 times with the same abrasive material, no fresh abrasive being added. Thus 11 segments are ground into the glass plate by only the amount of abrasive used for the initial cut. It was found that this number of repeated tests, when the best abrasive was used, resulted in a reduction of the ground area by about 5 pct. As this was a value that could be determined with sufficient accuracy and as a poor abrasive would produce a greater percentage reduction, 10 repeated tests were considered sufficient.

Fig. 3 is a photograph of three test plates made with three different abrasives. The left plate shows a series of tests on five portions, *A* to *E*, of a sample of synthetic corundum. The other plates show sim-

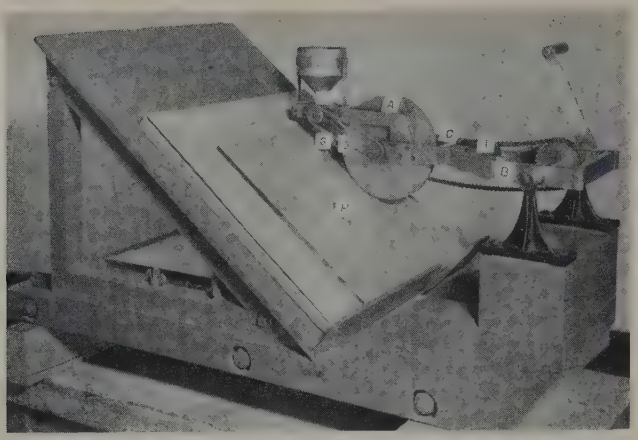


Fig. 2—National Bureau of Standards field or portable abrasion tester.

ilar tests on samples of low- and high-grade natural corundums. The areas of the first and eleventh segments are determined, and the percentage difference between the two segments is calculated for the abrasive. The result is a measure of toughness or breakdown value. In the preliminary studies the abrasives were collected after each test, and a sieve analysis was made for each sample.

Table I. Average Weight Losses of Abraded Specimens in the Standard Laboratory Abrasion Machine, with Various Abrasive Types

Type of Abrasive	Average Weight Loss, G	Tests, No.
Natural corundum	0.13	8 samples, 45 determinations
Artificial corundum	0.22	2 samples, 12 determinations
Silicon carbide	0.33	4 samples, 24 determinations

Table II. Average Weight Losses of Abraded Specimens in the Standard Laboratory Abrasion Machine, with Various Natural Corundums

Sample No.	Weight Loss, G
36	0.15
37	0.13
54	0.13
39	0.12
53	0.12
55	0.12
38	0.10

Results: Average weight losses of the abraded specimens in the standard laboratory abrasive machine are shown in Tables I and II. It is obvious from Table II that the tests using the standard laboratory abrasion machine were not sensitive enough to distinguish clearly between the abrasive properties of the various natural corundums.

The field abrasion tester produced results indicated in Table III. These tests were indicative of the relative hardness of the various types of abrasives. Results of breakdown tests with the field tester, as previously described, are shown in Table IV.

Of the two methods employed to test the relative hardness of the abrasive samples, the one using the field abrasion tester seemed to be the more efficient and practical. This machine was comparatively inexpensive to construct and is simple to operate. It requires less than 1 min to grind a segment on any suitable standard material, such as plate glass. A value of average area, S_x, as calculated from five segments cut by each sample under test, is sufficient for evaluating the relative hardness of the sample.

The performance of the field abrasion tester gave reproducible results in a short period and with a minimum of material and effort. Also, it appeared that values of segment area abraded, S_x, obtained from the abrasive value test and the percentage of decrease in segment area, D_x, obtained from the grain breakdown tests could be precisely specified in comparison with similar values from a synthetic corundum (Al_2O_3) chosen as the standard abrasive for these tests. These values are most simply expressed as the ratios: S_x/S_{std} and D_x/D_{std}. In this manner specific yet practical techniques were developed for grading natural corundum according to those physical characteristics for which it is used by industry. Consequently, there were incorporated into the final specifications minimum ratio values of S_x/S_s and of D_x/D_s for each grade of crystal and crystal concentrate. Detailed directions for obtaining these values, following the tests developed with the field abrasion tester, were also included.

Determination of Actual Corundum Content

The problem of determining the actual corundum content of an ore was simultaneously investigated by the Physical and Chemical Section of the Eastern Experiment Station, Bureau of Mines. This Bureau had been interested in corundum evaluation for some time. During the period from 1918 to 1942 no marketed production of corundum had been reported in the United States. Active interest in corundum was revived when World War II created a new and pressing demand for this abrasive material. To locate domestic supplies and thus reduce the dangers and uncertainties of a long ocean haul, an intensive search of areas formerly producing corundum, as well as possible new sources of commercial deposits, was undertaken by the Bureau of Mines and the Geological Survey in cooperation with the War Production Board. Deposits of corundum in Georgia, North Carolina, South Carolina, Montana, and other states were re-explored.

At this time numerous samples of corundum ore were received by the Bureau of Mines for ore-beneficiation and mineralogical studies. A representative cut of each sample was given to the petrographic laboratory for a quantitative determination of the corundum content. This procedure usually involved crushing the sample through a No. 70 screen, heavy liquid separation in methylene iodide, sizing of the light and heavy fractions on No. 100, 200, and 325 screens, identification of all the minerals present, and a determination, by petrographic grain counting, of the percentage of corundum in each size fraction. Corundum would also be determined quantitatively by this technique in numerous concentrates and tailing products submitted by ore-dressing metallurgists of the station.

During this same period, additional samples of foreign corundum ores, submitted for potential stockpiling, began to be received from other Federal agencies. The Bureau of Mines was requested to examine these ores and to determine whether they would meet the Munition Board's Material Purchase Specifications for corundum. Bureau of Mines petrographers were surprised to find that these existing specifications did not require a determination of the actual percentage of corundum, and they were at a complete loss in attempting to determine

what percentage of a sample had a scratch hardness equal to or better than 9 on Mohs scale. This hardness specification was, of course, wholly impractical.

The Bureau representatives suggested that a more realistic specification would be one embodying a quantitative determination of the mineral corundum rather than an assumed corundum content based essentially upon an Al_2O_3 determination.

Table III. Average Area Abraded into Plate Glass by Rotating Wheel of Field Abrasion Tester

Type of Abrasive	Average Area of Segment $(S_x)_{mm^2}$	Tests, No.
Natural corundum	4.31	8 samples, 24 determinations
Artificial corundum	6.68	2 samples, 6 determinations
Silicon carbide	8.76	3 samples, 12 determinations

In the course of these investigations members of the staff of the petrographic and the chemical analytical laboratories developed what is now called a *leach-petrographic* method to determine corundum.

The acid-leaching procedure is essentially that designated the "Fraser Reid Method, modified" by the American Cyanamid Co. and kindly furnished the Bureau of Mines by Bruce D. Crawford of the American Cyanamid ore-dressing laboratory.

A representative sample of the original ore is obtained by coning and quartering followed by further reduction of size with a Jones-type sample splitter. A −60 +100 mesh representative fraction of this material is selected for the leach-petrographic test. This size fraction is used to conform as closely as practicable to the No. 80 grain size used in the National Bureau of Standard tests. Frequently the sized fraction was examined for the presence of tramp iron by a hand magnet, although careful crushing usually avoided this contamination. The details of this leach-petrographic method are as follows:

1—Place 18 of No. 80 grain material in a large platinum crucible; add 15 cc of strong sulphuric acid and 20 cc of hydrofluoric acid. Place on the water bath or sand bath and evaporate until all the HF is expelled; finally, heat until fumes of SO_3 come off copiously.

2—Transfer to a beaker and add 20 cc of hydrochloric acid. Place on the hot plate and heat for 20 min to dissolve all soluble sulphates. Dilute to 75 to 100 cc with hot water, and heat 10 min longer. Filter and wash first with hot 5 pct HCl, then hot water, then 3 times with warm 10 pct KOH, then again with hot water. Ignite. Weigh as corundum.

Fig. 3—Plate glass test plates. Segments ground with various abrasives with the portable abrasion tester. Left, abrasive value and grain breakdown tests of synthetic corundum, standard grade; center, of natural corundum, high grade; and right, of natural corundum, low grade.

3—Examine the residue with the petrographic microscope for the presence of other insoluble minerals such as kyanite and rutile. If these are present, a petrographic grain count is made of the residue to obtain the corrected value. The count is made as follows: First, a few milligrams of the sample are mounted on a glass slide in a 1.740 refractive index oil and covered with a glass cover slip. In the microscope the corundum grains are readily distinguished by the blue rim surrounding them. All grains with marked negative or positive relief are gangue minerals. Second, by means of a laboratory integrating counter, the corundum content is determined by a count of corundum grains against all others. This is done by making a series of horizontal traverses and counting the grains touching the horizontal crosshair. Each horizontal traverse is spaced by a vertical interval of 1 mm. One thousand grains are counted in each mount.

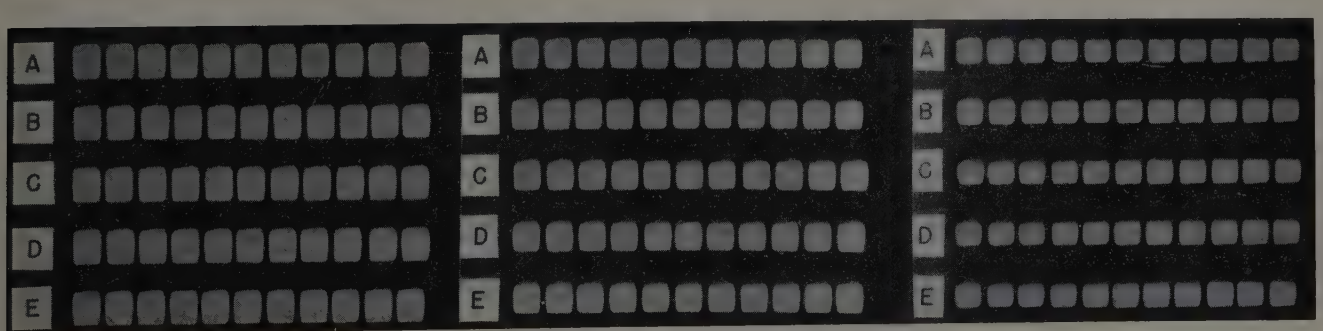
A detailed description of the petrographic method of fragment counting is given by F. Chayes in a series of three papers.[1-3]

Table IV. Break-down Characteristics of the Abrasives

Sample No.	Amount Passing No. 120 Sieve, Pct Originally	After 11 Tests	Decrease in Segment Area After 11 Tests (D_x) Pct
		Company A	
57 Al_2O_3	0	19	5.2
58 SiC	2	21	6.4
56 SiC	2	26	8.2
		Company B	
61 Al_2O_3	5	38	15.4
60 SiC	0	24	5.8
59 SiC	1	27	6.1
		Natural Corundum	
35	1	41	9.6
36	0	62	18.2
37	1	65	20.4
38	1	59	18.1
39	2	65	23.0
53	1	45	13.8
54	1	47	10.4
55	1	88	29.5

The petrographic examination also is used to detect the presence or absence of twinning, inclusions, well-developed rhombohedral parting planes, and incipient alteration. The influence of these factors has not been fully assessed, and opinions differ as to their actual effect on abrasive qualities of the ore. Of course, alteration to diaspore and to micaceous minerals and serious crystal defects would show up in the abrasive tests.

In many cases the weight of the insoluble residue from the acid leach gives directly a reliable index of the corundum percentage. This applied particularly to many of the African corundum ores tested, which contained few insoluble minerals other than



occasional grains of green spinel. However, this is not always the case. One particular sample of Indian corundum ore produced 85 pct of insoluble residue after the acid leach. A petrographic examination and a grain count of the residue showed that it contained only 49 pct corundum. The remaining 36 pct was composed of kyanite and rutile, both minerals being insoluble in the acid treatment.

Table V. Comparison of Petrographic Analyses and Hardness Tests on Natural Corundums

Sample No.	Insol., Pct	Corundum, Pct	Std. Lab. Tester, G	Field Tester, S_x	Breakdown Test, Pct	Breakdown D_x
35 Africa	91.7	90.8	0.21	5.05	41	9.6
36 Africa	61.7	61.7	0.15	4.10	62	18.2
37 Africa	77.7	77.7	0.13	3.52	65	20.4
38 Africa	80.6	80.6	0.10	3.72	59	18.1
39 Brazil	69.2	69.0	0.12	4.98	65	23.0
53 India	89.9	89.9	0.12	4.98	45	13.8
54 India	90.6	90.6	0.13	4.90	47	10.4
55 India	84.8	49.4	0.12	3.19	88	29.5

Other samples studied were found to contain sillimanite, diaspore, and spinel in the insoluble residues of the acid leach. (It might be noted here that the leach-petrographic method may also be used for determining kyanite and sillimanite in their ores.) The use of an acid leach alone, without subsequent petrographic examination of an ore containing appreciable amounts of diaspore (Al_2O_3 = 85 pct) or of kyanite (Al_2O_3 = 62 pct), would lead to a very incorrect value of the corundum content. Two samples from Bangalore, India, showed Al_2O_3 contents of 80.6 pct and 86.2 pct while the complete leach-petrographic examination indicated only 53 pct and 77 pct corundum, respectively. In both cases, the discrepancy represented appreciable amounts of kyanite.

The eight samples of natural corundums tested by the National Bureau of Standards were also tested for corundum content by the Bureau of Mines leach-petrographic method. The results are indicated and compared in Table V.

It is interesting to observe that the results of the breakdown test compare favorably with the percentage of corundum determined by the leach-petrographic method. Note that the sample, No. 55, having the poorest (highest) values of D_x and of breakdown showed the greatest divergence in values of percentage of insoluble and percentage of actual corundum. Those samples with the best (lowest) D_x have the least leach-petrographic discrepancies. Likewise, the samples having the highest actual corundum content gave the best values of both S_x and D_x.

Also note that although samples No. 39-Brazil and No. 53-India both have similar values of S_x, area ground, or *abrasive hardness,* they have different values of D_x, resistance to breakdown. This indicates the importance of the determination of D_x.

Table VI. National Stockpile Materials Specifications—P 18, Dated 3 January 1951, for *Corundum, Natural Crystals**

Grade	Minimum Corundum, Pct	Size	Minimum Ratio Values S_x/S_s	Minimum Ratio Values D_x/D_s
A crystal	87.5	Not over 5 pct —3 mesh	0.78	0.52
B crystal	85.5	Not over 5 pct —7 mesh	0.68	0.42
C crystal	80.5	Not over 5 pct —7 mesh	0.58	0.32
A crystal conc.	85.5	Approx. ¼ in. to 16 mesh and not over 5 pct —20 mesh	0.73	0.47
B crystal conc.	83.5		0.63	0.37
C crystal conc.	80.0		0.53	0.27

* Copies are available from the Emergency Procurement Service, General Services Administration, Washington 25, D. C.

Other Methods for Determining Corundum

One leading abrasive company uses a specific-gravity determination as a convenient means of evaluating the corundum content of an ore. This method is not entirely satisfactory and can be misleading, as the impurities can also have a high specific gravity, for example, rutile (4.24) and kyanite (3.6). If both minerals were present in equal amounts, their average specific gravity would be 3.92, a value similar to that obtained for many corundum-ore samples. Obviously, specific-gravity measurements without accompanying petrographic examinations are little better than Al_2O_3 determinations in assessing the amount of actual corundum present in an ore.

Further work is necessary to evaluate the degree to which various corundums are attacked by the acids used in the leach test. Sized samples of several grades of corundums, including gem-quality material, should be submitted to leach tests to determine to what degree and at what grain size various grades of the mineral are appreciably attacked by acids. Little or no solubility of *sound* grains is to be expected in the size range of —60 +100 mesh (approximately No. 80 grain size). However, fines of —200 or —325 mesh size and material containing cracks, inclusions, and alteration products may be partially dissolved by strong acids.

Likewise, further studies should be made on material that passes a certain grade by specific-gravity measurements but fails by the leach-petrographic tests. Perhaps abrasive tests on such samples would be conclusive. Complete quantitative mineralogical grain counts on various sizes should also be of value in determining the true quality of such ores.

Final Results

In summary, the final revised specifications for *corundum, natural crystals,* dated January 1951, include establishment of size requirements for both crystal and crystal concentrates, see Table VI. The percentage of corundum, rather than of alumina, for three grades of each type are specified, and definite abrasive characteristics are stated in detail. In addition, specific instructions are incorporated concerning the extraction of a representative sample, the preparation of the test sample, the leach-petrographic test for determining actual corundum content, and the method of performing an abrasive-value test and a grain-breakdown test.

To date, the Bureau of Mines and the National Bureau of Standards have been making their respective tests for evaluating samples submitted by the Federal Supply Service of the General Services Administration, and clear-cut evidence has been obtained for the acceptance or rejection of the submittals to the Goverment's program of corundum-ore stockpiling.

References

[1] F. Chayes: Petrographic Analysis by Fragment Counting—Part I. *Economic Geology* (1944) **39**, No. 7, pp. 484-505.

[2] F. Chayes: Petrographic Analysis by Fragment Counting—Part II. *Economic Geology* (1945) **40**, No. 8, pp. 517-525.

[3] F. Chayes: A Correction Factor for Specific Gravity and Volume Differences in Fragment Analysis. *Economic Geology* (1946) **41**, No. 7, pp. 749-760.

Suspension Preheating of Dry Pulverized Materials

by G. K. Engelhart

A multi-stage counterflow process developed in Germany preheats dry pulverized portland cement raw materials held in suspension in rotary kiln waste gases. Capacity of the first kiln installed for this purpose in the United States has been increased from 860 to 1500 bbl per day, with fuel reductions from 1,100,000 Btu to 650,000 or 700,000 Btu per bbl. Sixty percent of total waste gas is used. As full use of the gas stream is approached, additional kiln production and fuel economy may be expected.

EFFECTIVE use of rotary kiln waste gases to preheat dry pulverized materials has been demonstrated in full-scale operation at the Allentown Portland Cement Co. plant, Evansville, Pa. Pulverized raw materials are preheated in suspension in the gas stream in a multi-stage counterflow process which draws the gases through a series of cyclone collectors. Upon entering the first cyclone stage the material, continuously delivered and suspended in the gas stream, is successively collected and resuspended until it discharges through the kiln feed pipe. Gas and material contact time is thus prolonged, providing a high degree of heat recuperation with a very low temperature differential between gas and material at the final stage.

The suspension preheater, developed by Klockner-Humboldt-Deutz AG of Cologne, Germany, was first installed at the Norddeutsche Huette plant, Oslebshausen, Germany. Similar preheaters are now in operation at three other German cement plants. The first American installation, at the Allentown Portland Cement Co. plant, was designed and manufactured by Fuller Co. of Catasauqua, Pa., Humboldt's licensee for the United States, Canada, and Mexico.

Design of this installation is essentially the same as the German original, but provision was made for varying the method to adapt the process to specific objectives of American portland cement production. Differences between German and American cement clinker production practices are directly related to differences in three primary production cost items: fuel, labor, and power. Because fuel costs are high in Germany and elsewhere in Europe, thermal efficiency has been the first objective of European cement producers and manufacturers of rotary kilns. The Germans have developed such recuperative equipment as the present suspension preheater, the Miag calcinator, and the Lepol kiln, the invention of O. G. Lellep.

In North America labor is the principal cost item, although rising fuel costs are of growing concern. High clinker production is therefore of first importance. Recent installations of longer dry process cement kilns have been disappointing, failing to make satisfactory reductions in exit gas temperatures when production is held at an acceptable level. The Humboldt suspension preheater satisfies both

G. K. ENGELHART is Vice President, Fuller Co., Catasauqua, Pa.

Discussion on this paper, TP 3766H, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, Nov. 20, 1953. New York Meeting, February 1954.

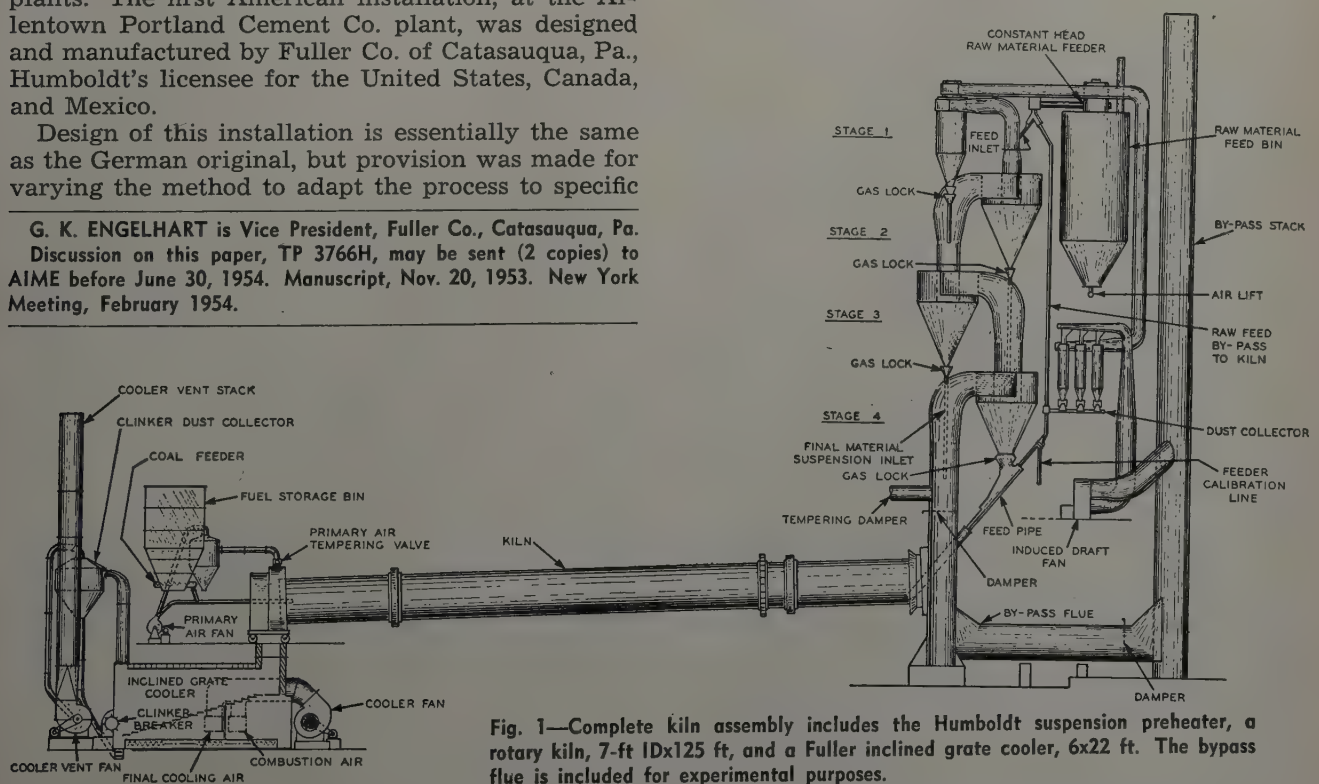


Fig. 1—Complete kiln assembly includes the Humboldt suspension preheater, a rotary kiln, 7-ft IDx125 ft, and a Fuller inclined grate cooler, 6x22 ft. The bypass flue is included for experimental purposes.

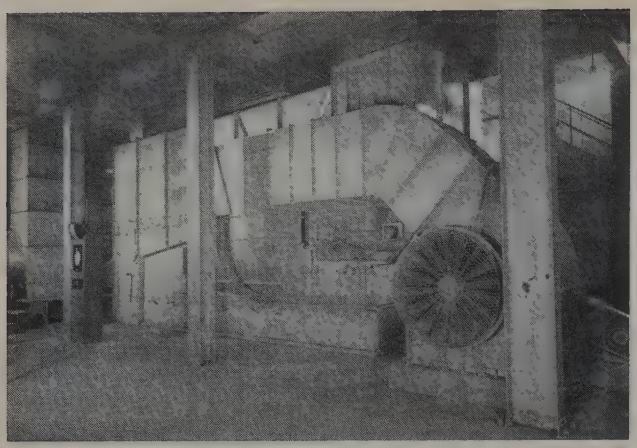


Fig. 2—A view of the clinker cooler, showing branches of the cooling air duct, which supply kiln combustion air and final cooling air.

objectives, greatly increasing production, relative to kiln volume, and lowering fuel consumption per barrel of clinker produced. It should extend the useful life of the many short kilns operating in North America.

Dry Raw Materials

The raw material supply of the Allentown Portland Cement Co. is an argillaceous limestone of the Jacksonburg formation, typical of the Lehigh Valley bluestone but somewhat higher in silica ratio than average. High-grade limestone from the company quarry near Oley, Pa., is blended with the basic material, and is mixed with iron oxide, when required, before grinding. Ultimate composition correction and contact is accomplished by mixing in silos. Normal fineness is 90 pct −200 mesh.

Raw materials from the plant of an associated company, the Valley Forge Cement Co., were burned during a full-scale experiment. Chemical composition of the feed was substantially the same but mineral content was slightly different. The mixture was prepared by dry grinding and blending rather than by normal beneficiation process, which includes a step of froth flotation.[1,2] No significant differences developed in thermal effects or clinker quality, and data included in this discussion are therefore limited to the Allentown Portland Cement Co. material.

Neither of the raw materials mentioned above contains kaolin or other fine or colloidal material. Attempts to nodulize these and other Lehigh Valley cement raw materials for preheating by other methods failed because adhesive characteristics were unsatisfactory.

Arrangement of Kiln

Complete kiln assembly, Fig. 1, comprises the Humboldt suspension preheater, a 7-ft IDx125 ft rotary kiln, and a 6x22-ft Fuller inclined grate cooler. For experimental purposes installation includes a bypass flue whereby all the kiln exit gases, or any desired proportion, can be diverted directly to the stack. To determine the capabilities of the preheater operation was restricted for the first five weeks to kiln and inclined grate cooler only, with all kiln gases exhausting directly to the stack. This is conventional practice, except that stack and bypass flue were designed for preheater operations rather than for continuous operation of the kiln without preheater.

For purposes of this discussion, the cooler should be referred to as a recuperator of clinker heat, capable of reducing total fuel consumption as much as 15 pct. Clinker discharging from the kiln, preferably at a temperature of about 2500°F, is distributed as a bed several inches thick on alternate fixed and reciprocating grates. The motion of the grates maintains clinker particles under continuous agitation as the bed moves toward the cooler discharge point. A plenum chamber below the grates is divided to assist in diverting most of the kiln combustion air through the hottest clinker. Most of this air is used as secondary combustion air. Preheated primary air is withdrawn from the top of the kiln hood, the location of minimum turbulence, and tempered with atmospheric air to a safe temperature. The balance of the cooling air, exceeding that which can be used for combustion, is forced through the lower portion of the bed to reduce clinker temperature below 150°F, rendering it suitable for grinding. This air is exhausted to atmosphere through a vent stack. A neutral or low negative pressure is desired in the chamber above the bed, and balance between combustion air and exhaust air is maintained by automatic vent stack damper control.

The elevated temperature of 2500°F for clinker discharged from the kiln, the result of burning as close to the nose ring as practical, permits abrupt air quenching to freeze a maximum of the liquid phase compounds (about 25 pct of the weight) to undercooled liquid or *glass*, although this practice increases available kiln volume and promotes flame development. A side elevation, Fig. 2, of the clinker cooler shows branches of the cooling air duct, which supply kiln combustion air and final cooling air, respectively. The connection to the kiln hood, through which the clinker falls and air passes to the kiln, extends the full width of the cooler. The balanced crank drive for the grates is shown below the air duct. The 10° pitch of the grates, Fig. 3, is indicated by the flange of the brick casing between the observation door and the building column. The clinker breaker is shown at extreme left.

Kiln and cooler assembly, without the preheater, produced an average of 860 bbl of clinker a day at a fuel consumption of 1,100,000 Btu per bbl. This is very satisfactory for conventional kilns of this size.

Fuel supply, Fig. 4, at this plant is unusual, comprising a mixture of 77 pct anthracite, 13 pct bituminous coal, and 10 pct oil. The oil is used to promote ignition of the anthracite and may be omitted if the proportion of bituminous coal is increased.

Fig. 3—The 10° pitch of the grates in this view of the clinker cooler is indicated by the flange of the brick casing between the observation door and the building column. The clinker breaker is shown at left.

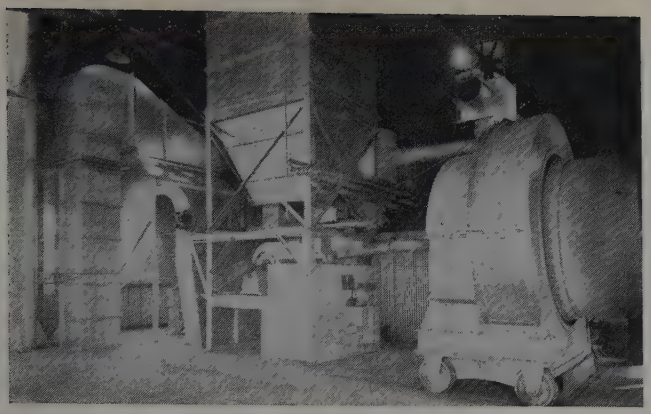


Fig. 4—The fuel supply system is shown above.

The kilns are bin fed, and anthracite and bituminous coal are ground together in a single tube mill. The primary air connection, with its tempering valve, may be seen above the kiln hood; air is drawn through the small cyclone collector to protect the primary air fan. The pulverized fuel mixture is delivered to the burner pipe by a screw feeder of usual type. The Buell low-resistance collector in the background separates fine clinker particles from final cooling air vented to atmosphere.

The Humboldt Suspension Preheater

The Humboldt suspension preheater, Fig. 1, was started at Evansville, Pa., late in May 1953. The preheater includes a raw material supply bin; a series of four cyclone collector stages, with gas ducts; a dust collector; an exhaust fan and the usual stack. Exit gases from the kiln are drawn upward through a vertical duct having a short lateral, opening tangentially into the final or fourth stage of the preheater. Similar ducts deliver the gas to the third and second stages, but the gas stream is divided and passes from stage 2 to two cyclones of narrow diameter which form stage 1. These cyclones are provided to increase collection efficiency. From stage 1 the gases pass through a dust collector and fan to the stack.

Raw material is continuously delivered to the system by a Fuller airlift-constant head feeder. This feeder is calibrated and its speed is adjustable from the burner floor. It delivers a stream of constant unit weight, discharging centrally into the duct leading from stage 2 to stage 1 at a point near the bottom of the duct.

All gas duct dimensions are calculated to provide adequate velocity at the temperature required to suspend the material and carry it vertically to the cyclones. After separation in stage 1 the collected material passes through balanced gates, which serve as gas locks, and is conducted by tubes opening into the gas duct between stages 3 and 2. The preferred point of entry is immediately above the discharge opening of stage 3.

Stages 2, 3, and 4 are essentially identical, the material being alternately suspended and collected, finally discharging into the kiln feed pipe. Time of gas and material exposure from initial suspension in stage 1 to final discharge from stage 4 is approximately 20 sec, sufficient time for transfer of heat.

Dust escaping in the exhaust ducts of stage 1 is collected by four banks of three high efficiency cyclones. Final dust lading in the stack discharge is 3 grains, after 40 pct of the gas is bypassed. Collected dust discharges into the kiln feed pipe in combination with the discharge of stage 4. Mechanical interferences prevented convenient return of collector dust to the riser duct between the kiln and stage 4. Since this dust amounts to about 5 pct of total kiln feed, and since its temperature averages 245°F, it should be returned to the system for preheating, preferably to stage 4, as a closed circuit. This change is designed and is expected to effect further fuel economy.

Construction and arrangement of the preheater and raw material supply systems are illustrated in Fig. 5. The relation of this assembly to the kiln is shown in Fig. 6. To improve initial recuperation, the back (feed) end of the kiln was provided with lifters for a distance of 12 ft, comprising brick 4 in. square elevated 3 in. above the surface of the lining on 1-ft centers.

A tempering valve was designed to admit atmospheric air into the kiln exhaust duct to provide for temperature control, if calcination proved undesirable or if heated materials became sticky, building up in collector discharge ports. Some tendency to stickiness, particularly in the discharge from the final stage, was overcome by increasing the diameter of the balanced gate.

All cyclones and ducts below stage 1 are lined with cast refractories 6 in. thick. Stages 2, 3, and 4 have an ID of 9 ft 10 in. Each of the two cyclones forming stage 1 has an ID of 5 ft 3 in.

Table I is based upon operation with only 60 pct of total kiln gas conducted through the preheater, the balance being discharged through the bypass directly to the stack.

The most significant data are the relative temperatures of gas entering and material discharging from stage 4 and the temperature of the fan intake compared with kiln exit gas temperature.

Clinker capacity rose from 860 to 1500 bbl per day and fuel consumption dropped from 1,100,000 to between 650,000 and 700,000 Btu per bbl.

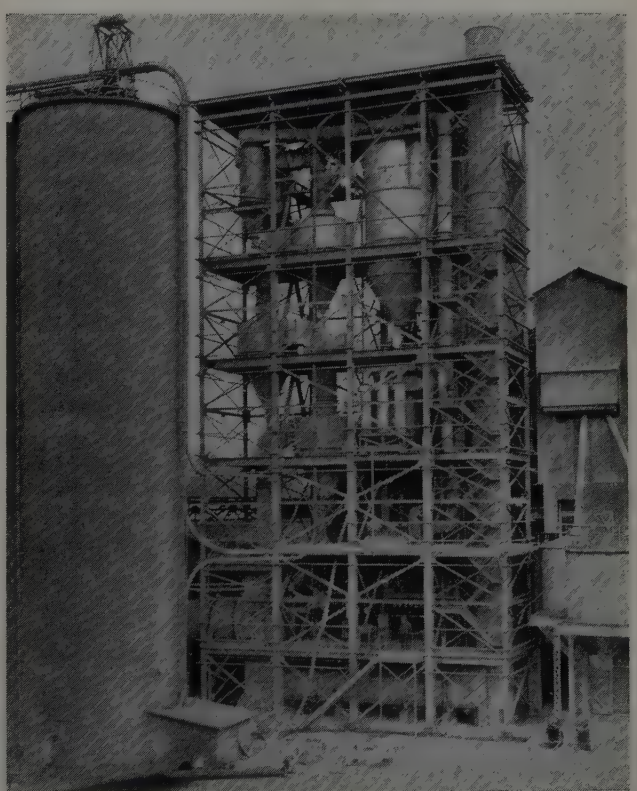


Fig. 5—Arrangement of preheater and raw material supply systems.

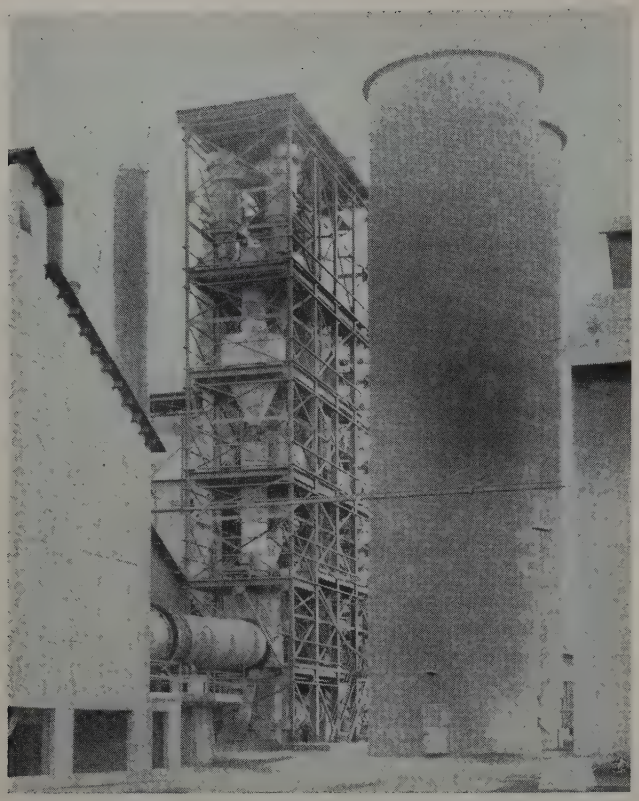


Fig. 6—Relation of preheater and supply systems to kiln.

Concurrently with the recording of heat transfer data, tests were conducted to determine whether or not this method would have adverse effects on the quality of cement clinker and, presumably, on the products of other pulverized materials treated in rotary kilns. These tests included investigations of possible segregation of constituent minerals because of particle size distribution or differences in specific gravity, aggravated, perhaps, by calcination of carbonates in the lower stages of the preheater. Calcination occurs, but reliable data are not available to determine what part of total ignition loss may be attributed to calcined particles carried into the system from the kiln. Segregation has not been detected, however. It was found unnecessary to return the dust collector discharge to the raw feed or gas streams, but this would undoubtedly increase total recuperation. It is assumed that repeated collection and continuous flow account for maintained blend and contact.

Table I. Comparative Gas and Material Temperatures at Principal Preheater Change Points, 60 Pct Gas to Preheater, Balance to Stack, Average of 41 Tests

Item	Temperature, °F
Kiln, waste gas (preheater operating)	1986
Kiln, waste gas (without preheater)	1565
Stack gas, combined	615
Bypass flue	920
Gas, duct, stage 1 to dust collector	535
Gas, inlet to fan	465
Gas, duct, kiln to stage 4	1504
Material, stage 4 to kiln	1332
Gas, duct, stage 4 to stage 3	1260
Material, discharge stage 3	1190
Gas, duct, stage 3 to stage 2	1016
Material, discharge stage 2	946
Gas, duct, stage 2 to stage 1	667
Material, discharge stage 1	590
Static Pressures Throughout Preheater, Water, In.	
Duct from kiln to stage 4	1.6
Duct from stage 4 to stage 3	5.2
Duct from stage 3 to stage 2	9.6
Duct from stage 2 to stage 1	14.1
Duct from stage 1 to collector	18.4
Duct from collector to fan	22.4

Another possible adverse effect was in ultimate discharge of gases from the system at temperatures below condensation temperatures of the alkalies. As compared with clinker of the same raw material composition, produced concurrently in another kiln of the plant, average analyses of 18 days determinations, by flame photometer, were as follows. Average theoretical total alkali, expressed as Na_2O in feed to the kilns, was 1.036 pct. In the short kiln, without preheater and without flue dust collection and return to the feed, the total alkali in the clinker was reduced from 1.036 to 0.53 pct, a reduction of 48.5 pct from the theoretical limit. From the kiln, with preheater and flue dust recovery and return, alkali was reduced from 1.036 to 0.623 pct, a reduction of 40.1 pct from the theoretical limit.

This slight gain would not justify discarding part of the collector dust of highest alkali concentration. Clinker produced is consistently of high quality, improved density, and lower free lime as compared with clinker produced by the other four kilns at the plant.

Proposed Experiments

Low temperature differentials between material and gas, particularly at the third and fourth stages, appear to confirm early estimates that four stages may not be necessary to obtain satisfactory thermal efficiency. As this is written, the preheater is being modified to permit delivery of raw material directly to any stage. The cyclones of the present installation will continue to carry the gas stream, thus serving as part of the dust collection system. One apparent advantage of the four-stage preheater, apart from its proved heat exchange capacity, is that it discharges gas at temperatures within the efficient operating range of electrical dust precipitators. The first stage and its related duct work can be made of black steel without refractory lining.

Conclusions

Suspension preheating is an efficient method for increasing capacity of dry process cement clinker kilns. It appears to have a potential for fuel reduction superior to other known methods. With the possible exception of existing kilns equipped with waste heat boilers, suspension preheater installations for existing kilns are economically justifiable. Fuel savings would justify suspension preheaters for dry short kilns over long kilns. On the basis of relative fuel economy, wet process kilns of adequate length, equipped with modern chain systems, have an apparent advantage over wet process kilns equipped with suspension preheater installations preceded by slurry or filter cake dryers, since the heat content of preheater exhaust gases is too low to complete drying. For existing dry process kilns, preheaters should be designed so that the only capacity limitation is the practical fuel-burning capacity of the kiln. Neither calcination in the preheater nor occasional secondary combustion in the preheater presents operating problems.

References

[1] B. L. Miller and C. H. Breerwood: Flotation Processing of Limestone. AIME TP 606-H.26 (February 1935).

[2] G. K. Engelhart: *Industrial and Engineering Chemistry* (May 1940) **32**, p. 645.

Solid State Bonding in Iron Ore Pellets

by Strathmore R. B. Cooke and Robert E. Brandt

For a study of the bonding that occurs in magnetite pellets during oxidation, cubes of magnetite, hematite, and quartz were prepared, each with one surface polished and nearly optically plane. These faces were fired together under pressure and at various temperatures to determine whether bonding would occur. Sections of surfaces that bonded were examined microscopically. An attempt was made to measure the strength effected by this treatment.

EARLIER work[1, 2] has shown that the strength developed by fired magnetite pellets containing silica or silicates is due to three separate effects. Two of these are associated with the formation of slag in the interior of each pellet at temperatures above 1000°C, where the atmosphere is substantially though temporarily neutral. The third is independent of the silica content and is responsible for the development of a hard shell around the pellet during the initial stages of firing, a condition which permits the pellet to assume the increasing load of the overlying burden and prevents extrusion of the slagged and semi-liquid interior. Pellets made from hematite concentrates do not show these effects unless specific additives are incorporated, and it is these differences in behavior which in large part account for the difficulty in producing satisfactory pellets from such concentrates.

Elsewhere it has been stated that "the most important factor responsible for the development of strength in fired magnetite pellets containing no additive is the oxidation of magnetite to hematite and the subsequent recrystallization and grain growth of this constituent."[1] The object of the investigation reported in the present paper was to study in detail the mechanism described above and to evaluate its contribution to the strength of the fired pellet compared with other possible mechanisms, excluding slag bonding. This was effected by placing together polished faces of two cubes prepared from specific minerals and then heating them under constant pressure at different temperatures both in air and in nitrogen.

The materials used were magnetite, hematite, and quartz. The magnetite was coarsely crystalline material from western Montana, the hematite was Itabira ore from Brazil, and the quartz was of optical grade but of unknown origin. A high-grade Lebanon (Pennsylvania) magnetite concentrate was used to prepare briquets for the investigation of bonding strength. Analyses of the iron oxide materials are given in Table I.

The specimens for furnace treatment were prepared with a carborundum cut-off wheel, which cut the minerals into cubes approximating 3/8 in. along each edge. Care was taken to make the sides as parallel as possible. The edges of the faces to be polished were beveled, and the cubes were mounted in brass rings with sealing wax, this being essential to prevent the specimens from rocking during polishing. To prevent excessive friction, the wax was recessed so that only the brass ring and the face of each specimen would be in contact with the polishing lap. Each specimen was then ground with No. 600 carborundum to produce a level surface before polishing.

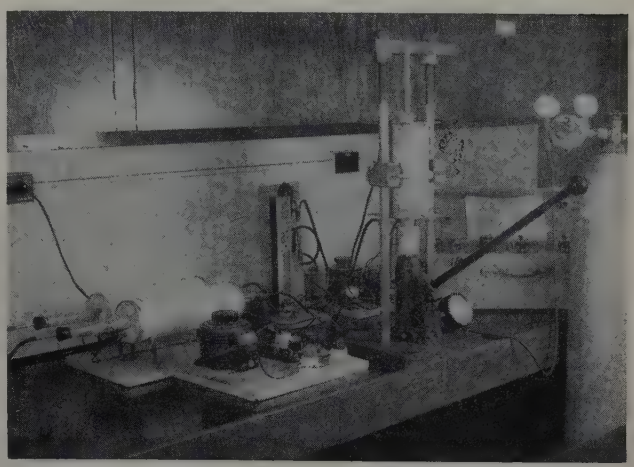


Fig. 1—Furnace for firing specimens under pressure and in a controlled atmosphere.

Table I. Analyses of Iron Oxide Materials

Magnetite Concentrate, Pct		Montana Magnetite, Pct		Brazilian Hematite, Pct	
Fe, total	71.34	Fe, total	66.05	Fe, total	69.94
Fe++	23.86	Fe++	18.06	Fe++	0.40
SiO_2	0.52	SiO_2	2.26	SiO_2	0.05
		MgO	3.25		
		CaO	0.04		

The Graton-Vanderwilt process was not used for polishing because of the long time required by this method, but several other methods were tried. Cloth laps using levigated alumina or chromium oxide did not produce the desired polish. Satisfactory results were obtained by means of a pitch lap,[3] which was used for all the mineral cubes, with levigated alumina as the polishing medium. The lap was operated in a machine similar to those used in the preparation of single optical surfaces. It was run at low speeds and moderate pressures, and polishing was stopped occasionally to prevent overheating of the lap. A good polish with flat surfaces could be obtained in about 5 min. After polishing the sealing wax was removed either in hot water or in a solvent such as turpentine.

S. R. B. COOKE, Member AIME, is Professor of Metallurgy and Mineral Dressing, School of Mines, University of Minnesota, Minneapolis. R. E. BRANDT, Student Member, AIME, is now with the M. A. Hanna Co., Hibbing, Minn.

Discussion on this paper, TP 3745B, may be sent (2 copies) to AIME before June 30, 1954. Manuscript Sept. 24, 1953. New York Meeting, February 1954.

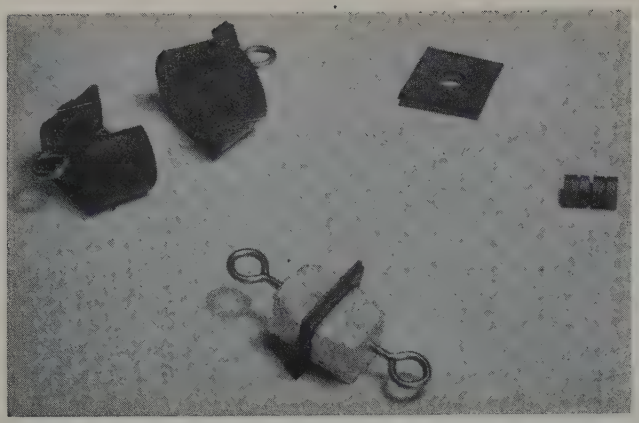


Fig. 2—Method of mounting briquets to obtain tensile strength.

An electric tube furnace, Fig. 1, was designed to fit between the uprights of an hydraulic press and mounted so that it could be raised or lowered at will. The specimens were placed on a movable ¾-in. 18-8 stainless steel rod attached to the hydraulic jack. Pressure was applied to the specimens by raising the assembly against a similar rod fixed to the upper frame of the press. The furnace, which was built around the pressure rods, was then lowered to center the specimens in the hot zone. The firing temperature was controlled with a Variac voltage regulator and was measured by a thermocouple passing through the furnace wall and resting against the specimens at their contact. A maximum of 1000°C was obtained in this apparatus.

Modifications of this equipment were made when a neutral atmosphere was desired. Fig. 1 shows the final design for operating in a controlled atmosphere. An electrically heated reservoir was built around the lower pressure rod and was filled with molten Wood's metal. An air-tight seal could then be made by lowering the furnace so that the silica tube was immersed in the molten bath. Nitrogen was admitted through copper tubing extending through the bath and into the furnace from opposite sides. It was found that commercial nitrogen contained a considerable amount of oxygen. This was removed by passing the gas over heated copper turnings. The thermocouple was introduced through the top of the furnace tube, and the free space was plugged loosely with glass wool to avoid eddy currents of air which would sweep back into the furnace. This arrangement worked satisfactorily, and no oxidation could be detected microscopically in the fired specimens.

In operation, the furnace and pressure rods were pre-heated to the desired firing temperature. The furnace was then raised so that two specimens with their polished faces in contact could be placed on the lower pressure rod. Care was taken properly to align the specimens, and a slight pressure was applied to hold them in place. The furnace was then lowered around them and the thermocouple placed in position. The specimens reached firing temperature in about 7 min. A pressure of approximately 1000 psi was then applied to hold them in intimate contact, and this was held until the end of the run. The firing time was measured from the time the specimens reached temperature until the end of the run. The furnace was then raised, thus permitting air-quenching.

The firing technique in nitrogen was different from that used in air, since it was impossible to maintain the atmospheric seal while charging the pre-heated furnace. The specimens were therefore placed in the furnace at room temperature and the atmospheric seal was established by lowering the furnace into the molten Wood's metal. Air was flushed out of the system with nitrogen at a flow rate of approximately 300 cu cm per min, and the specimens were heated to the desired firing temperature. A pressure of 1000 psi was applied and maintained until the furnace was shut off, but the flow of nitrogen was continued until the specimens had cooled to room temperature.

Those cubes which bonded during firing were mounted in sealing wax, sectioned across the junction, remounted in a copper ring with sealing wax, and polished with the pitch lap. Photomicrographs

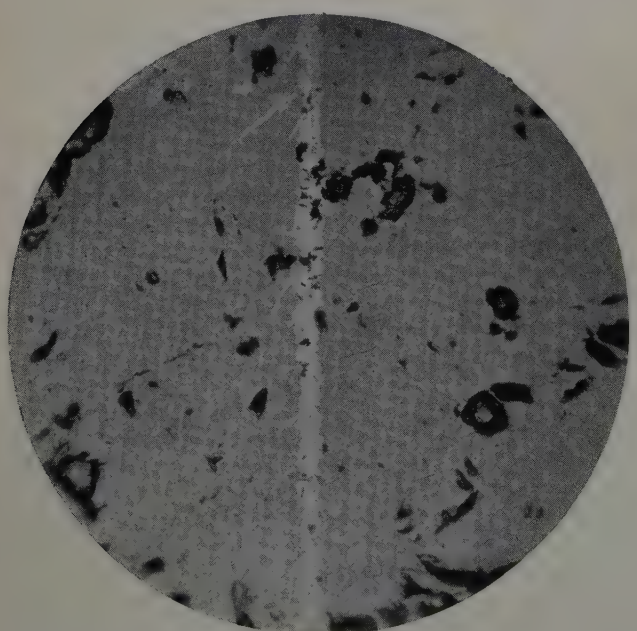


Fig. 3—Bonding between magnetite cubes fired in air for 4 hr at 700°C. X500. On the original print fine blebs of magnetite can be seen along the contact.

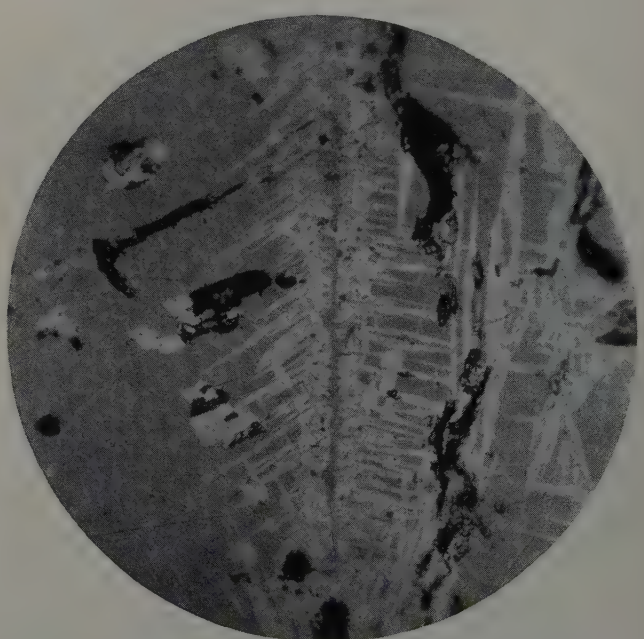


Fig. 4—The junction between magnetite cubes fired in air for 1 hr at 900°C. The magnetite along the contact is very pronounced. X500.

were then taken to record different contact structures obtained with various firing conditions.

An effort was made to measure tensile strengths of the bonded cubes, but because of the naturally occurring cracks in the minerals the data obtained were not reproducible.

In an attempt to get around this difficulty, *briquets* were prepared from the magnetite concentrate. Five-gram samples of —200 mesh concentrate containing 10 pct water were placed in a ½-in. steel mold and a pressure of 24,000 psi was alternately applied and released 10 times to ensure maximum compaction of the particles. The green briquets were then removed and placed in an electric furnace. To remove the water the temperature was raised gradually from room temperature to 450°C over a period of 2 hr. Firing was then effected by raising the temperature to 1300°C and holding at that temperature before furnace-cooling to room conditions. All furnace operations were performed in a nitrogen atmosphere.

The fired briquets were uniform to 1/1000 in. diam and appeared to be free from external or internal cracks. They could not be polished on a pitch lap because lap material was forced into the interstices, but they were coherent enough to take a good polish on a Graton-Vanderwilt machine. After preparation of one flat polished surface on each briquet by the last-mentioned method, a series of two briquets with polished faces in contact was fired in precisely the same manner as previously described for the mineral cubes.

A special procedure, shown in Fig. 2, was adopted to determine tensile strengths of the briquets which bonded under these firing conditions. Peripheral notches were cut in the specimens, and a rubber sleeve was placed over the plane of contact. The specimen with its sleeve was then placed in a mold containing two steel screw eyes so disposed that they lay in a straight line perpendicular to the plane of contact. Molten Wood's metal was then poured around the specimen. When this solidified each end of the specimen was firmly gripped by metal which had entered the notches. The tensile strength of the bond was determined by breaking in an Amsler testing machine.

The strengths of individual briquets were also measured by this method. Here the briquet, approximately 1 in. long, was deeply notched at its center, as shown in Fig. 2, to provide a zone of weakness. Otherwise the method of mounting and of testing was the same as described above.

The area of fracture in both cases was measured by projecting its image on a ground glass plate and tracing on paper the fractured area in relation to the total area of cross-section of the briquet. The ratio of areas was determined from the weights of the cut papers representing the relative areas. Tensile strength was reported on the basis of kilograms per square centimeter.

Experimental Results

Magnetite-Magnetite Fired in Air: Magnetite cubes in contact were fired in air at temperatures ranging from 500° to 1000°C for different times. The lowest temperature at which a bond strong enough to withstand sectioning and polishing was obtained was 500°C, and in this case it was necessary to fire the specimen for 8 hr. At 600°C strong bonding occurred after 4 hr of firing. One hour of firing produced weak bonding below 700°C, insufficient, however, to permit subsequent handling. It is evident that both the time and the temperature are important factors in the bonding of these magnetite cubes.

Figs. 3, 4, and 5 show typical structures developed along the plane of bonding. At low and moderate temperatures the bond consists of a narrow band of hematite in which there is dispersed a small amount of magnetite, which may or may not be residual. At high temperatures, 900° and 1000°C, and with prolonged heating, the oxidation spreads out into the surrounding magnetite to a pronounced degree, and a prominent band of magnetite develops along the original junction, see Figs. 4 and 5. Occasionally a continuous field of magnetite extends across the contact, see Fig. 6.

Fig. 5—Bonding between magnetite cubes fired in air for 4 hr at 1000°C. The magnetite along the contact is strongly developed. X500.

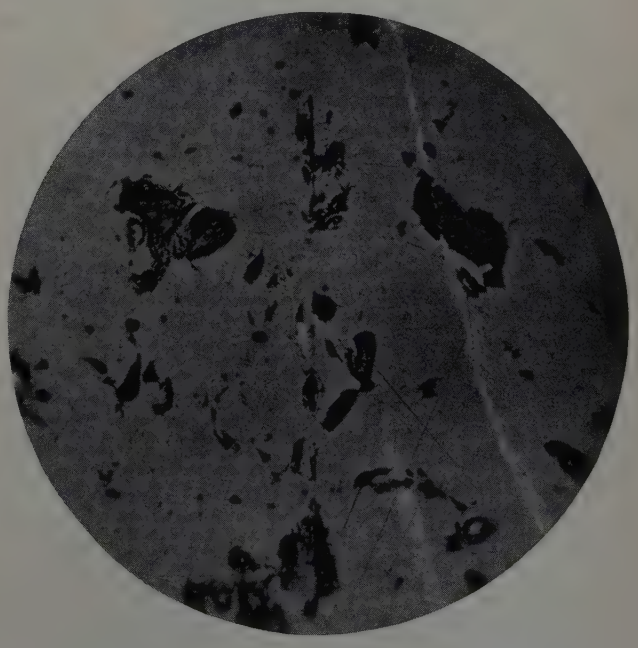


Fig. 6—Photomicrograph of another portion of the junction of the same specimen shown in Fig. 3. The contact here is magnetite, with no trace of hematite. X500.

Magnetite-Magnetite Fired in Nitrogen: No bonding occurs between magnetite cubes fired in nitrogen at temperatures up to 1000°C.

Magnetite-Hematite Fired in Air: Bonding takes place at 600°C after 4 hr of firing, but not at lower temperatures, and at 800°C after 1 hr of firing. With commensurate firing times, the bonding takes place at a higher temperature than that of the magnetite-to-magnetite system. Fig. 7 shows that the bonding can be attributed to the cementation of the freshly-formed hematite to the original hematite. Magnetite, which is apparently residual, may also be prominent along the contact, see Fig. 8. However, in Fig. 9 the magnetite extends as embayments across the contact into the hematite.

Magnetite-Hematite Fired in Nitrogen. No bonding occurs in the magnetite-to-hematite system fired in nitrogen.

Other Systems Investigated. Magnetite and quartz do not bond in either air or in nitrogen at temperatures up to 1000°C. This is to be expected, since Bitsianes has shown[4] by X-ray study that solid reaction between magnetite and quartz takes place at 900°C only after firing for a very long time, approximately 20 hr.

No bonding is obtained between hematite and quartz or between hematite and hematite when fired in air at temperatures up to 1000°C.

Tensile Strength Measurements. Tensile tests made on bonded *cubes* failed to give reproducible data. Unfortunately, in spite of many precautions made during testing, heat-bonded polished *briquets* also failed to give reproducible tensile strengths.

Another attempt was therefore made to evaluate the tensile strength effected by cementation resulting from the oxidation of magnetite. This was done by firing green briquets, prepared as described before, in air for 3 hr at temperatures ranging from 800° to 1400°C. Tensile strengths were determined as described under Experimental Procedure. Results are shown in Fig. 10, each point representing the arithmetic mean of the strength of six briquets. Maximum strength was reached at a temperature of 1200°C, with rapid decline at higher temperatures. Minute cracks could be seen in those briquets fired at 1400°C, a condition which may have been incipient at 1300°C, and which could account for the decrease in strength. The measured apparent density decreased above 1200°C, and it should also be pointed out that appreciable quantities of hematite convert to magnetite at 1300°C or higher.

To evaluate the tensile strength caused by recrystallization of magnetite, another series of briquets was fired in nitrogen and tested. The results are also given in Fig. 10. These briquets possessed inappreciable strength below 950°C, but it rose rapidly above that temperature to a maximum at 1300°C. The apparent density increased steadily from 950° to 1400°C.

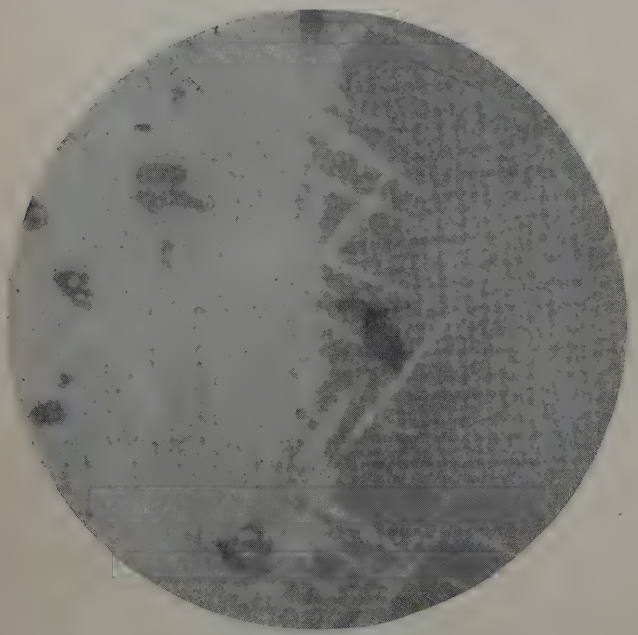


Fig. 7 (upper left)—Bond between magnetite and hematite cubes fired in air for 4 hr at 700°C. X500. The hematite is the white constituent.

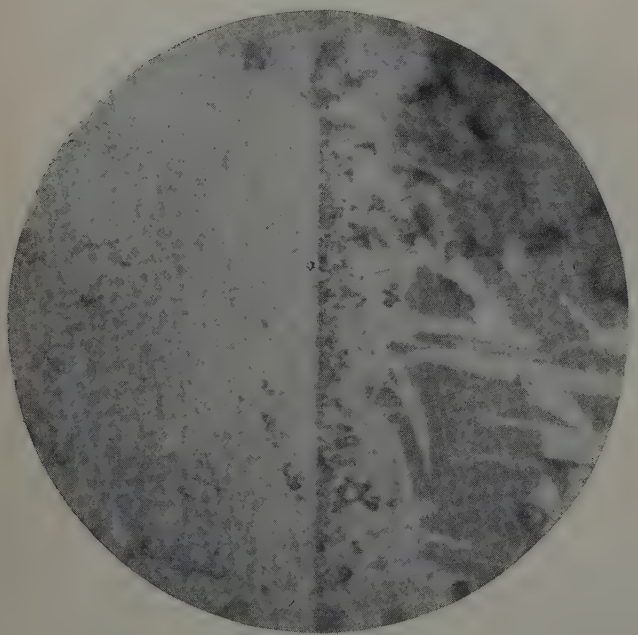


Fig. 8 (lower left)—Bond between magnetite and hematite cubes fired in air for 4 hr at 800°C. X500. Note the magnetite along the contact.

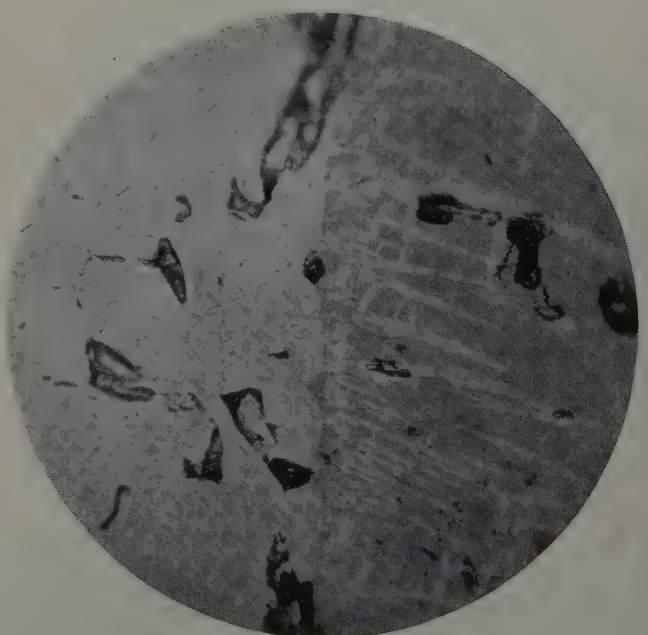


Fig. 9 (lower right)—Junction between magnetite and hematite cubes fired in air for 1 hr at 1000°C. X500.

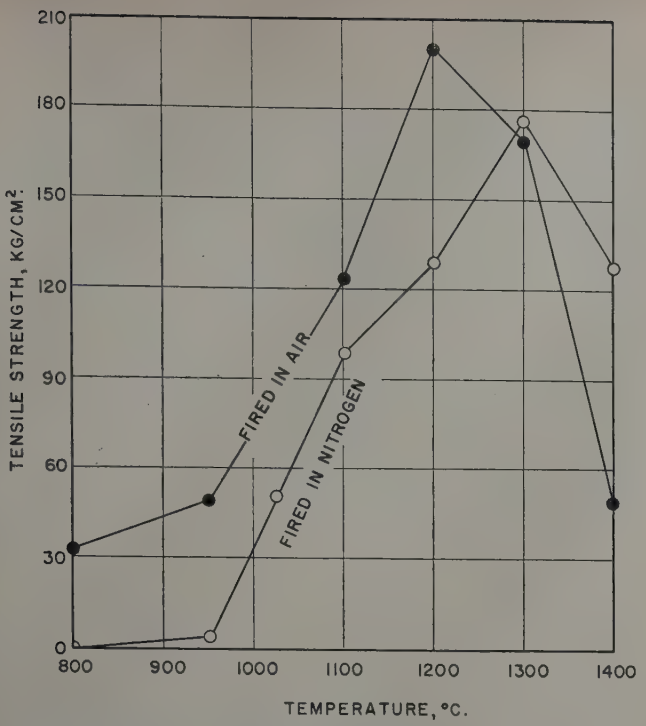


Fig. 10—Tensile strength vs temperature curves for magnetite briquets heat-treated for 3 hr.

In this investigation it was found that in a neutral atmosphere direct bonding of magnetite-to-magnetite cubes or of magnetite-to-hematite cubes, within the limits of pressure and time investigated, does not occur at 1000°C or lower, and that direct bonding of magnetite particles does not occur to any extent below 950°C. Above this temperature recrystallization, manifested as interparticle bridging, causes the tensile strength of briquets to rise rapidly to a maximum, as shown in Fig. 10. Compressive strength data,[5] obtained by crushing pellets made from the same material as the briquets and fired in nitrogen, gave similar results, demonstrating good correlation of strengths obtained by two different methods of testing.

The strength of briquets fired in air at temperatures up to 1300°C is greater than that of briquets fired in nitrogen. This is an apparent contradiction of data presented elsewhere,[2] which shows that the strength of pellets fired in nitrogen is greater than that of pellets fired in air above 1000°C. In this last-mentioned case the high silica content of the pellets gave rise to a duplex structure, materially lowering their compressive strength. Cooke and Stowasser,[1] however, have shown that if magnetite pellets are pre-oxidized and then re-fired at higher temperatures, the crushing strength becomes greater than that of similar pellets fired in nitrogen. The strength of briquets fired in air, see Fig. 10, resembles these data for pre-heated pellets..

Cementation of two magnetite cubes under oxidizing conditions occurs, under pressure, at a temperature as low as 500°C. The effect of pressure was not investigated in detail, but at high temperatures bonding occurs between polished magnetite faces held together at very low pressure. A study of a large number of sections shows that the degree of cementation increases with the temperature and with the time of firing. Crystal orientation of adjacent surfaces is not a factor governing cementation. Bonding magnetite-to-hematite in air requires higher temperature than magnetite-to-magnetite.

Fig. 6 shows the interesting and not unusual phenomenon of an apparent magnetite-to-magnetite bond formed at 700°C in air. This could be either a magnetite bond or a cementation bond. If the former, it may have been caused by unusually good contact and high unit pressure at that point, for the polishing technique did not permit optically plane surfaces to be formed on the cubes. It is probable, however, that this is not a direct bond because this type of bonding did not occur when similar cubes were fired in nitrogen at temperatures up to 1000°C. A better explanation is that cementation of areas of very close contact has occurred in the usual manner, followed by solid solution of the small amount of hematite in the surrounding magnetite.

When cubes of magnetite are fired together in air at low temperatures, blebs of magnetite are frequently found along the original line of contact and completely enclosed by hematite, see Fig. 3. These may be residual magnetite. At higher temperatures, however, there seems to be a definite migration of hematite away from the junction and a corresponding replacement by magnetite. This becomes very pronounced at 1000°C, see Fig. 5.

Conclusions

This paper presents a study of the solid state reaction responsible for the development of the hard shell in conventionally fired magnetite pellets and the high strength and uniform internal structure in laboratory pellets pre-oxidized before final firing.[1] The conclusions are summarized as follows:

1—Bonding occurs between magnetite cubes fired in air at temperatures as low as 500°C. The degree of bonding increases with temperature and time.

2—Bonding occurs between magnetite and hematite cubes in air at higher temperature, starting at about 600°C.

3—No bonding is effected between magnetite cubes or between hematite and magnetite cubes when fired in nitrogen at temperatures up to 1000°C. This clearly indicates that oxidation governs the bonding mechanism at this or lower temperatures.

The bonding effected by heat treatment in air is caused by conversion of magnetite to hematite, the hematite forming a practically continuous layer between the cubes. At high temperatures the hematite tends to migrate away from the bond and to be replaced by magnetite.

5—Owing to many uncontrollable variables, it was found impossible to determine quantitatively the strength of the bonding, although an extensive study of tensile strengths was made. However, a tensile method of determining the strengths of briquets was developed which gave data comparable to those obtained by compression tests on pellets made from the same material and heat-treated in the same manner.

References

[1] S. R. B. Cooke and W. F. Stowasser: The Effect of Heat Treatment and Certain Additives on the Strength of Fired Magnetite Pellets. *Trans.* AIME (1952) **193**, p. 1223.

[2] S. R. B. Cooke and T. E. Ban: Microstructures in Iron Ore Pellets. *Trans.* AIME (1952) **193**, p. 1053.

[3] S. R. B. Cooke: Microstructure and Concentratability of the More Important Iron Ores of the United States. U. S. Bur. Mines *Bull.* 391 (1936).

[4] Private communication.

[5] T. E. Ban: The Agglomeration of Fine Taconite Concentrates by Pelletizing. Master's Thesis, University of Minnesota, June 1951.

Flotation of Oxidized Zinc Ores

by M. Rey, G. Sitia, P. Raffinot, and V. Formanek

Six years of laboratory study, followed by three years of mill operation treating more than 10,000 tons of ore, have established the flotation of oxidized zinc ores with fatty amines as an efficient process. Recently the method has been applied with success to partially oxidized sulphide ores. In this paper the authors give an account of their research and the milling results obtained in Sardinia since 1951.

CONCENTRATION of oxidized copper and lead ores by flotation has been practiced for 30 years, but flotation of oxidized zinc ores has remained unsolved until a few years ago.

This problem is, however, of great importance in mining districts where large tonnages of oxidized zinc ores exist in underground mines or in dumps. Some sulphide ores also possess a degree of oxidation high enough to justify recovery of oxide zinc minerals. Various attempts have been made to float the zinc carbonate, smithsonite.

It is well known[1] that smithsonite can be collected with fatty acids, but as the predominant gangue constituents, limestone and dolomite, are floatable with the same reagents, fatty acids are difficult to use. Gaudin has shown,[2] on mixture of pure minerals, that smithsonite can be floated with higher xanthates, or better, with higher mercaptans. Another procedure, briefly mentioned by Davis,[3] consists in sulphidizing the ore, activating the zinc carbonate with copper sulphate, and floating with ordinary xanthates. This process was attempted in the writers' laboratory in 1946, but results were erratic. It has since been taken up by Italian engineers and is in operation on certain types of ores.

Two other research projects should be mentioned. In 1942 Erlenmeyer and others[4] floated synthetic mixtures of smithsonite and gangue minerals with hydroxyquinoline, an analytical reagent for zinc. On the other hand, Bunge, Fine, and Legsdin published in 1946[5] some interesting results obtained on Missouri oxidized zinc ores. With sodium oleate as a collector and a combination of sodium hydroxide, sodium silicate, and citric acid as a limestone depressor, they secured, after four cleaning operations, a good grade of concentrate. With many ores this reagent combination lacks adequate selectivity.

In 1939 M. Rey initiated a program of research on this important problem. Work was pursued first at the School of Mines in Liège, Belgium[6, 7] and later in Paris in the laboratories of Société Minière et Métallurgique de Penarroya and Société Minerais et Métaux. Reagents used in the beginning were higher xanthates and mercaptans, later fatty amines. Sulphidization with sodium sulphide was found necessary. These conditions make possible the flotation of zinc carbonate and zinc silicate.

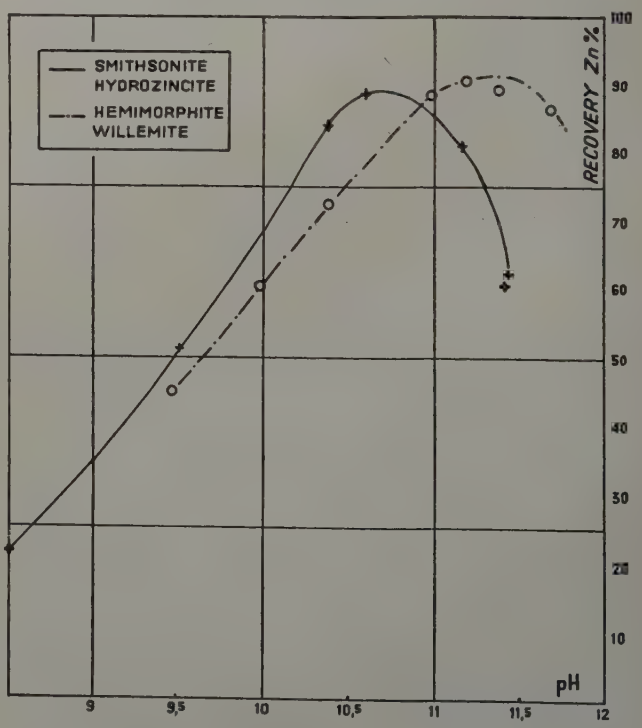


Fig. 1—Relationship between pH and recovery of oxidized zinc minerals.

Two milling plants were put in operation in Sardinia in 1950 and several others are now contemplated.[8, 9]

It should be mentioned that unknown to the present writers McKenna, Lessels, and Peterson were

M. REY, Member AIME, is Professor and Director of Flotation Research, Societe Miniere et Metallurgique de Penarroya Paris. G. SITIA, Member AIME, is Director of Mines, Societa Mineraria e Metallurgica di Pertusola Genoa, Italy. P. RAFFINOT and V. FORMANEK are Research Engineers, Societe Minerais et Metaux, Paris.

Discussion on this paper, TP 3768B, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, Nov. 23, 1953. New York Meeting, February 1954.

working on the same problem and in 1949 were granted a U. S. patent[10] covering a reagent combination similar to the one which was used by M. Rey and his associates.

Oxidized zinc ores usually contain the zinc-bearing minerals indicated in Table I. The carbonates occur chiefly in ores having a limestone or dolomite gangue. Hemimorphite is abundant when the gangue is rich in quartz but the carbonates and the silicate are often present together. Willemite is exceptional. The degree of association of the minerals with the gangue and the character of the gangue are exceedingly variable. Limestone, dolomite, quartz, talc, clay, and various iron oxides and hydroxides are found. In many instances a 100-mesh grind gives satisfactory liberation of zinc minerals, but in others this is not the case and the degree of association present is extremely fine. This is particularly so with brown or ferruginous calamines, which are sometimes completely unsuitable to flotation or any mechanical treatment.

Table I. Zinc-Bearing Minerals

Mineral	Formula	Zinc, Pct
Smithsonite	$ZnCO_3$	52.1
Hydrozincite	$ZnCO_3.Zn(OH)_2$	60.5
Hemimorphite	$Zn_2SiO_3(OH)_2$	54.2
Willemite	Zn_2SiO_4	58.0

The presence of slime, composed of more or less ferruginous clay, is also in certain cases a problem, as is the presence of soluble salts, particularly calcium sulphate.

As a general rule it can be stated that the lighter the ore color, the greater the chance to treat it with high recovery and low reagent consumption.

More than 50 samples of ore belonging to 30 different mines have been investigated so far in the authors' laboratory. Of these 90 pct have responded favorably to treatment. These samples came from the following countries: France, Italy, Spain, Sardinia (Italy), Tunisia, Algeria, Morocco, French Congo, Norway, and Southwest Africa.

Technique of Amine Flotation of Oxidized Zinc Ores

Normal procedure is to float first the lead sulphide and oxide minerals, and the sphalerite, if present, with the usual reagents. Tailing from the lead or sphalerite flotation is the feed to the new flotation process. It is also possible to float sphalerite and oxidized zinc minerals in one operation and to separate these later if desired.

Flotation of oxidized zinc minerals comprises three steps: conditioning, sulphidizing, and addition of collecting and frothing agents. In the following discussion these steps will be considered separately.

Effect of Slimes. Conditioning: As is well known, slimes have a marked effect on flotation with amine reagents, owing to the tendency of amines to adsorb on their surface. When this happens the concentrates become contaminated, flotation of granular material is impaired, and reagent consumption is increased.

Frequently, however, flotation can be carried out in the presence of the slimes if these are conditioned and neutralized in the right way. This may be done by use of sodium carbonate and sodium silicate, polyphosphates (Calgon), and some organic protective colloids, such as starch and carboxymethylcellulose.

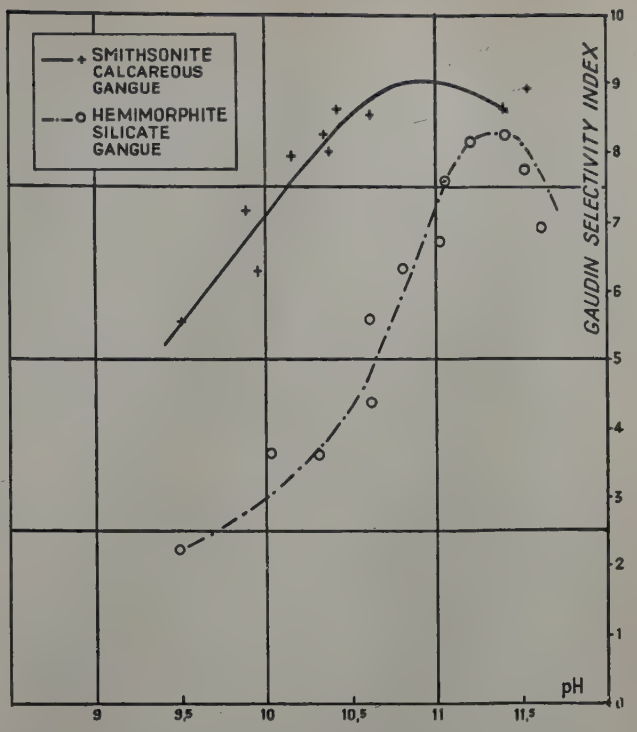


Fig. 2—Relationship between pH and Gaudin selectivity index.

In certain cases slimes are so noxious that it is better to deslime the ore before flotation. The desliming operation may be carried out with cyclones, and in extreme cases a double desliming may be practiced to obtain a very clean feed for flotation. The desliming operation is also necessary when ores are of low grade or when the price of zinc is low, since in that case flotation of the slime portion of the ore is not economic. The slimes usually have about the same zinc content as the ore; accordingly the metal loss usually ranges from 10 to 20 pct.

Sulphidizing. Effect of Sodium Sulphide: After conditioning or desliming or both, sodium sulphide is added and the amine collector immediately after, together with a frother. Action of the sulphide differs entirely from its action on lead and copper carbonate. With these minerals an excess of sodium sulphide acts as depressant, and flotation starts only when this excess has been consumed by the ore. Hence the necessity of a certain conditioning time. In amine flotation of the zinc minerals, an excess of sodium sulphide has no depressing effect, on the contrary, and conditioning is not necessary. Addition of an alkali sulphide is indispensable. The authors have not succeeded in obtaining a selective float of the zinc minerals without its use.

The alkali sulphide acts first as a pH regulating agent, giving an alkaline pH to the pulp. Recovery and selectivity increase with pH, see Figs. 1 and 2, and best results are obtained between pH 10.5 and 11, although lower values are often admitted as a matter of economy.

The beneficial effect of a high pH suggests that the effective collector is not an amine salt or amine ion but a free amine RNH_2. An equilibrium exists between the two forms such as

$$R\text{--}NH_3^+ + OH^- \rightleftharpoons RNH_2 + HOH$$

and an increase in alkalinity liberates more free amine from its salt. This is in agreement with the

findings of Arbiter, Kellogg, and Taggart[11] and Kellogg and Vasquez-Rosas[12] in their studies of the action of amines on metallic zinc and sphalerite. Action of sodium sulphide in this type of flotation is not entirely clear and warrants further studies.

Collecting Agents. Amines: More than 50 different cationic reagents have been tested in the authors' laboratory. The conclusion is that quaternary ammonium salts, amides, and secondary and tertiary amines have no collecting power. Correct results are obtained only with primary amines.

Among these, the aliphatic amines are excellent, whereas aromatic amines such as aniline, toluidine, xylidine, and benzidine show no collecting power. Rosinamine, derived from rosin or abietic acid, can be used but its effect is impaired by its excessive sensitivity towards slimes.

The cause of collection may be sought in complex formation with the zinc ion similar to complexes with ammonia. This hypothesis has been put forward by Arbiter, Kellogg, and Taggart[11] and previously by Gaudin, in the study of the flotation of sphalerite. In the present case the idea is supported by the fact that all the zinc minerals are collected by the amines and by the fact that ammonia and ammonium salts act as depressants. The stability of complexes decreases in the following order: ammonia; primary, secondary, tertiary amine. This would explain the fact that only the primary amines act as collectors.

The amines are used in the form of soluble salts such as the acetates or hydrochlorides. Amine bases can also be used in emulsion or dissolved in an organic solvent.

Frothing Agents: Froths obtained with amines alone are in general not satisfactory and frothing agents such as pine oil, alcohols, and Dowfroth are usually employed.

Type of Flotation. General Results: When conditioning reagents, pH, pulp dilution, sodium sulphide, and type of amine reagent and frother are correctly combined, flotation is rapid and froths are heavily mineralized. They are usually of a cream or slightly brownish tinge, and after one or two stages of cleaning, grades of 40 to 45 pct zinc can be obtained.

In certain cases where the zinc minerals are finely disseminated in the gangue and liberation by grinding is incomplete, concentrates will contain a maximum of 38 to 39 pct zinc.

Grade of tailing may vary from 0.5 to 3 pct according to grade of heads and difficulty of concentration, so that recovery varies from 50 pct to over 90 pct in different cases.

Milling Plants in Italy, France, and French Morocco

After some preliminary tests in February 1950 two flotation sections were put in operation in the mills of Societa Mineraria è Metallurgica di Pertusola in Southern Sardinia, September 1950. Sardinia is known for its long record of production of calamines or oxidized zinc ores and the process is of great interest there. The above-named company was the first to use it on a commercial scale.

At San Giovanni, in the Iglesias district, 300 tons per day of various dump materials are treated. The mill will later treat oxidized zinc ore from the mine, set aside for years because its ore was too disseminated to be amenable to hand picking and jigging.

At Buggerru, 20 miles away (old Malfiano mine), ore is preconcentrated in a galena sink and float plant and the sink and fines are floated at the rate of 120 tons per day.

A small operation on dump material has been carried out in France for nearly a year.

A new development, 1952 to 1953, at Societa di Pertusola has been the flotation of oxidized zinc minerals from partially oxidized sulphide ores. At the mines of San Giovanni and Buggerru 300 tons per day of these ores have been treated for a number of years for lead and sphalerite recovery. Tailings carried about 3 pct zinc in the form of carbonate. These tailings are now floated at Buggerru, and final tailings have been brought down to around 1 pct. A similar operation, under study for San Giovanni, is possible because the mining and fine grinding costs are borne by the recovery of lead and sphalerite; the flotation operation bears only its reagent and operating cost.

Since the beginning of 1953 tests have been under way in Morocco at the Touissit mine of Compagnie Royale Asturienne des Mines and the Zellidja mine of Société des Mines de Zellidja. In spite of the present low price of zinc an attempt will be made to install the process as a commercial operation.

Milling of Oxidized Zinc Ores At Buggerru

The oxidized zinc ore of Buggerru is a dolomitic limestone containing zinc carbonate and a small amount of lead carbonate. After being crushed to 2-in. and deslimed in a log washer, the + ⅛ in. is treated in a Huntington-Heberlein sink-and-float plant.

During 1949 and 1950, when flotation was not yet in operation, the plant treated rich ore running 13 to 14 pct in zinc and 1.5 to 2.0 pct in lead. Density of medium was adjusted at 2.85 to make a high-grade sink and a float running 3 pct zinc.

Table II. Flotation of Oxidized Zinc Ores at the Buggerru Mill

	Year 1952		September 1953	
Metallurgical Results, Pct	**Zinc**	**Lead**	**Zinc**	**Lead**
Heads	13.02	1.66	13.07	1.11
Lead carbonate concentrate	9.88	63.00	10.97	57.62
Zinc carbonate concentrate	40.50	1.54	44.44	1.31
Tailings	2.21	0.02	1.51	0.02
Recovery	86.40	73.30	90.0	81.7
Reagent Consumption, Lb Per Ton	**Zinc Circuit**	**Lead Circuit**	**Zinc Circuit**	**Lead Circuit**
Sodium carbonate	3.70	1.10	3.80	2.00
Sodium silicate	2.90	3.60	3.60	2.00
Organic colloids	0.54		0.20	
Sodium sulphide	13.00	2.20	6.00	2.38
Sodium cyanide		0.06		0.03
Amylxanthate		0.08		0.12
Amine acetate	0.66		0.23	
Pine oil	0.36	0.20	0.20	0.20

The sink, containing 39 pct zinc and 6 pct lead, all in oxidized state, was reground and floated for recovery of lead carbonate. The flotation tailing was the zinc concentrate. Not all ores are capable of giving such a high-grade product at this coarse size, but in this case the result was of great interest.

Since the start of flotation operations grade of heads has been lowered to increase mine reserves and density of medium brought down to 2.7. In this way grade of float has been lowered to 2 pct zinc and metal loss reduced. The amount of float discarded represents 50 to 60 pct of the weight of run-of-mine ore. The sink at 19 pct zinc and fines at 11.3 pct zinc are combined and go to flotation.

Daily capacity of the sink-and-float plant is 240 tons, for the flotation section 120 tons.

The flotation section receives the reground sink product and the slimes from a Dorr thickener. The thickener is operated with a slightly turbid overflow to discard the finest slimes, which represent about 5 pct of the weight of ore.

The ground pulp is conditioned with sodium silicate, sodium carbonate, and sodium sulphide and floated for recovery of cerussite. With heads at 1.6 to 1.7 pct Pb, the concentrate, after double cleaning, runs 59 to 60 pct and tailing 0.30 to 0.35 pct Pb.

Tailing from the lead flotation section is further conditioned with carbonate, silicate, and a small amount of organic colloids to disperse and neutralize slimes. Flotation of the zinc carbonate is carried out with the acetate of an aliphatic amine as collector and with sodium sulphide. Flotation cells used are positive-flow minerals separation machines.

The concentrate is cleaned twice and shipped to the electrolytic zinc plant at Crotone, Italy. Results have been satisfactory from the start and have improved constantly. Fig. 3 is a flowsheet of the plant.

Table II gives average results for 1952 and for September 1953. Reagent consumption, particularly of sodium sulphide, was high in 1951 and 1952 but decreased considerably in 1953, partly because of an increase in amount of slime discarded by the Dorr thickener.

The partly oxidized sulphide ore of Buggerru is complex, as the limestone contains some pyrite, as well as sulphides and carbonates of lead and zinc. These five different minerals are recovered in series in the order: galena, sphalerite, pyrite, cerussite, smithsonite. The ore is deslimed before flotation of pyrite to facilitate later flotation and decrease reagent consumption. At this point the slimes carry only 2 to 3 pct zinc in oxidized form and their treatment is not economical.

Treatment of Partly Oxidized Sulphide Ore at Buggerru

Pyrite flotation is really a scavenging operation by which a small amount of pyrite, 1.5 to 2.0 pct, is floated away to clean the pulp and facilitate flotation of cerussite. Reagent consumption is very small and at Buggerru amounts to a few grams per ton of ethylxanthate and pine oil. A small amount of zinc sulphate is also used to depress any residual sphalerite and recover it later in the oxidized zinc concentrate.

Cerussite flotation is remarkable, owing to low grade of heads and small reagent consumption. Feed to the circuit assays 0.25 to 0.30 pct lead and tailing 0.15 pct. Concentrate runs 50 to 60 pct. This operation pays well because reagent consumption is very small:

Reagent	Lb Per Ton
Sodium sulphide	0.4 to 1.0
Amylxanthate	0.02 to 0.03
Pine oil	0.04 to 0.06

This flotation step improves the grade of zinc concentrate, which otherwise would be contaminated with lead. Removal of cerussite is also favorable because it consumes sodium sulphide necessary for flotation of zinc carbonate.

After flotation of the cerussite, smithsonite is floated and cleaned twice. Results for September 1953, see Table III, show that when pulp is properly

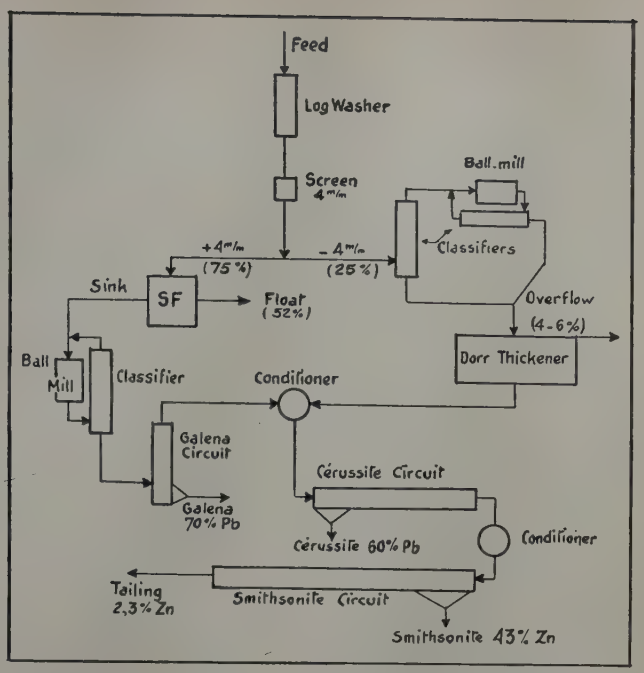


Fig. 3—Flowsheet for sink-float and flotation mill at Buggerru, Italy.

deslimed and cleaned of interfering minerals and when various factors such as pH, pulp density, reagent additions at various points of the circuit, and cleaning of concentrates are properly adjusted, oxidized zinc flotation can be carried out with small reagent consumption. The loss in cyclone overflow shown in Table III is high, but this is being remedied by better installation, and in October 1953 was already reduced to 26 pct. Cyclone overflow is lower grade than the underflow, a favorable factor.

Table III. Flotation of Partially Oxidized Sulphide Ores at Buggerru, September 1953. Pyrite-Cerussite-Smithsonite Circuits

Item	Weight, Pct	Assays: Total Zinc	Assays: Oxide Zinc	Assays: Lead	Recoveries: Total Zinc	Recoveries: Oxide Zinc	Recoveries: Lead
Feed	100.00	3.34	3.07	0.21	100.0	100.0	100.0
Cyclone overflow	34.5*	2.56	2.26	0.10	—	—	—
Cyclone underflow	65.5	3.56	3.21	0.26	72.5	72.9	83.5
Pyrite concentrate	1.45	14.15	1.96	2.48	—	—	—
Cerussite concentrate	0.16	6.00	6.00	57.14	—	—	43.8
Smithsonite concentrate	3.28	44.35	43.81	1.43	45.3	46.8	—
Tailing	60.61	1.09	1.04	traces	—	—	—

Reagent Consumption, Lb Per Ton of Cyclone Overflow

Pyrite Circuit	Lb Per Ton	Cerussite Circuit	Lb PerTon
Zinc sulphate	0.094	Sodium sulphate	0.890
Ethylxanthate	0.020	Amylxanthate	0.032
Pine oil	0.052	Pine oil	0.042

Smithsonite Circuit	Lb Per Ton
Sodium carbonate	1.770
Sodium silicate	1.340
Sodium sulphide	2.340
Amine acetate	0.100
Pine oil	0.100

* This figure is exceptionally high.

Operations at San Giovanni have been much more difficult, owing to the character of ore treated and the bad quality of water available.

So far the feed has been taken from dumps and has been highly ferruginous and clayey. The water available is hard and contains chlorides and sulphates that have a deleterious effect on flotation and increase reagent consumption.

Milling of Oxidized Zinc Ores at San Giovanni

The flowsheet includes flotation of cerussite with sodium sulphide and amylxanthate, then partial or total desliming with cyclones and flotation of oxidized zinc minerals, both zinc carbonate and zinc silicate.

During a certain period slimes from the cyclones have been reintroduced in the circuit at an appropriate point to increase metal recovery, but at present this is not done because of the low zinc price. On the contrary, thorough desliming is practiced before oxidized zinc flotation. The desliming operation cannot be carried out before lead flotation because, as is well known, there is always high concentration of lead minerals in the fine sizes. For a new mill, the advisability of desliming before fine grinding to reject the primary slimes would have to be considered.

Table IV. Flotation Results on Sands and Slimes, San Giovanni.

Item	+200 Mesh	200 Mesh to 10 Microns	−10 Microns
Weight, pct	79.20	15.77	5.05
Feed, Zn, pct	8.10	10.70	8.00
Concentrate, Zn, pct	43.50	45.20	22.20
Tailing, Zn, pct	1.30	1.50	3.85
Recovery, Zn, pct	86.50	88.80	62.70

The effect of slimes on metallurgical results, apart from the fact that they consume a large quantity of reagents, is shown by data in Table IV, obtained in sampling the flotation circuit and sizing the products, at a time when the desliming operation was not complete.

Results on the +10 micron feed are excellent but on the −10 micron slime are unsatisfactory. There is a net loss in treating the −10 micron part of the ore, as a 22 pct concentrate has a negative value, and as much of this material as possible must be removed before flotation.

Table V. Flotation of Oxidized Zinc Ores at the San Giovanni Mill, 1952

Metallurgical Results, Pct	Zinc	Lead
Heads	10.02	1.86
Oxidized lead concentrate	4.70	67.70
Oxidized zinc concentrate	40.90	1.60
Tailings	3.80	0.20
Recovery	69.50	78.30
Reagent Consumption, Lb Per Ton	**Zinc**	**Lead**
Sodium carbonate	4.500	1.870
Sodium silicate	5.220	11.640
Organic colloids	0.610	
Sodium sulphide	21.630	5.050
Amylxanthate		0.162
Petroleum	2.870	
Amine acetate	1.630	
Pine oil	0.320	0.104

Because of the difficulties described above, good metallurgical results have been obtained at San Giovanni only at the cost of a high reagent consumption. Table V shows results and reagent consumptions for 1952.

Reagent consumption is notably lower in 1953, chiefly owing to better desliming. Water purification is being studied and will also be helpful.

Concentrates from the San Giovanni mill contain some chlorine, due to the nature of the water, and cannot be used in an electrolytic zinc plant. They are calcined and shipped to a French smelter.

Sulphide ores treated at San Giovanni contain about 3 pct oxide zinc like those milled at Buggerru, and recovery of this part of the zinc is under study. Favorable results have been obtained in the laboratory and in pilot mill tests, but operations in the mill still leave much to be desired. The ore contains more pyrite than at Buggerru, one reason for the difficulties encountered.

Conclusions

After six years of study in the laboratory and three years of mill operation treating more than 100,000 tons of ore, it is held that flotation of oxidized zinc ores by the combination of sodium sulphide and primary amines is an efficient process and well under control. It has proved adaptable to a wide variety of oxidized zinc ores. Both mill and laboratory study of the numerous factors influencing results have been rewarded by gradual improvement in metallurgical results and gradual lowering of reagent consumption.

The process will certainly be of use in mining districts other than those where it is now carried out. The experience already gained, only partially detailed in this paper, will be invaluable.

Acknowledgment

It is not possible to mention all those who have labored to bring about success, but particular mention should be made of Messrs. Valdivieso, Cozza, Angotsi, and Mancuso of the staff of Societa di Pertusola. The authors also thank the firms Penarroya, Pertusola, and Minerais et Métaux for permission to publish this paper.

References

[1] Ralston: Flotation of Nonmetallic Minerals. U. S. Bur. Mines *R.I. 3397* (May 1938).

[2] Gaudin: *Flotation,* p. 315. McGraw-Hill Book Co., New York, 1932.

[3] Davis: Ore Testing Studies. U. S. Bur. Mines *R.I. 3370* (1936-1937) p. 99.

[4] Erlenmeyer, Von Steiger, and Theilheimer. Flotation avec la 8-hydroxyquinoleine. *Helv. Chim. Acta* (1942) **25**, p. 241.

[5] Bunge, Fine, and Legsdin. Missouri School of Mines *Bull.* (September 1946) **17**, No. 3.

[6] Gaty: Étude des Minérais Calaminaires Pauvres au Laboratoire de Métallurgie Spéciale. *Rev. Univ. Mines* 7 ème série, **29**, No. 5 (1942).

[7] Gaty: Étude de la Flottation des Minéraux Calaminaires. *Rev. Univ. Mines* 9 ème série, **2**, No. 4 (1946) p. 476.

[8] Rey and Raffinot: The Flotation of Oxidized Zinc Ores. *Recent Developments in Mineral Dressing.* Symposium, London (1952). French patents: 1.001.166 (1948-1952), 1.011.183 (1948-1952), 1.036.092 (1951-1953). Provisional: 634.568 (1952).

[9] Rey and Raffinot: La Flottation des Calamines. Congrès des Laveries des Mines Métalliques Francaises. Paris (1953). *Revue de l'Industrie Minerale* (1954).

[10] U. S. patent 2,482,859. Sept. 27, 1949.

[11] Arbiter, Kellogg, and Taggart: The Mechanism of Collection of Metals and Metallic Sulphides by Amines and Amine Salts. *Trans.* AIME (1943) p. 517.

[12] Kellogg and Vasquez-Rosas: Amine Flotation of Sphalerite-Galena Ores. *Trans.* AIME (1946) **169**, p. 476.

Leasing of Government Potash Lands

by H. I. Smith

WHEN Spain established colonies on the North American continent, some of her land grants, in what is now the United States, reserved to the Crown deposits of gold, silver, and mercury. Later mineral rights were reserved under some of the English Crown charters involving land in the eastern part of North America, with provisions for the payment of royalties thereon to the Crown.

Reservation of mineral deposits to the United States Government was instituted by an ordinance of Congress on May 20, 1785, which applied to such deposits in the Northwest Territory, then north of the Ohio River and east of the Mississippi River, and provided that there should be reserved "one third of all gold, silver, lead, and copper mines, to be sold or otherwise disposed of as Congress shall hereafter direct." Little was known then of the mineral resources of the country; the Great Lakes copper region had just come into the possession of the United States by treaty and much of the western mineral land still belonged to France and Spain.

The policy of leasing mineral deposits was enacted by Congress under the act of March 3, 1807, which provided that "the President of the United States shall be, and is hereby, authorized to lease any lead mine which has been or may hereafter be discovered in the Indiana Territory, for a term not exceeding five years," and in the same year the Government reserved 345,600 acres of land in northern Illinois, valuable for lead.

In 1816 Congress provided that in all cases where a tract of public land containing a lead mine or salt spring was applied for by settlers on the public domain, no permission to work the mine or spring would be granted without the approbation of the President of the United States.

By the act of March 1, 1847 (9 Stat. 146), the control of mineral lands was transferred, with all records, from the War Department to the Treasury Department, and by the act of March 3, 1849 (9 Stat. 395), supervisory powers over lead and other mines of the United States were transferred to the Secretary of the Interior.

Following the discovery of gold in California in 1848, President Polk advocated the leasing of mineral lands acquired from Mexico under the treaty of 1848. However, owing to the lack of communication and transportation facilities and the consequent difficulty of checking on production and operations, leasing was found impracticable. Mining was permitted to be carried on in each district under rules made by the miners themselves and patterned after the Mexican mining system. To meet the situation, Congress enacted mining laws in 1866, 1870, and 1872, which provided for possession by location and for private ownership, after discovery, by patent of essentially all mineral deposits belonging to the United States, except coal.

H. I. SMITH is Chief of the Mining Branch, Conservation Division, U. S. Geological Survey, Washington, D. C.

Discussion on this paper, TP 3723H, may be sent (2 copies) to AIME before June 30, 1954. Manuscript, Jan. 13, 1953. Los Angeles Meeting, February 1953.

Many other acts were passed by Congress applying to specific regions and states, reserving salt, lead, or other minerals, some of which provided for the leasing of such deposits. However, very few if any leases were issued thereunder.

Not until 1901 (31 Stat. 745) were the mining laws extended to include salt, and in 1910 they were revised to specify salines and associated products.

At the turn of the century, it became more and more evident that the mining laws developed for metallic minerals were not practical for the development and conservation of oil and gas, coal, potash, phosphate, or oil shale. Following withdrawals by the Secretary of the Interior as authorized by the act of June 25, 1910, the Department of the Interior recommended leasing legislation; however, it was not until February 25, 1920, that the Mineral Leasing Act was passed by Congress. This act authorized leasing all public lands potentially valuable for oil, gas, coal, phosphate, sodium, or oil shale.

The Organic Act, creating the Geological Survey in 1879, imposed upon its director the duty of classifying the public lands.[1] The early years of the Survey were devoted largely to the accumulation of fundamental data and, with only minor exceptions, land classification was not seriously undertaken until 1906. Since that year, it has been actively pursued with respect particularly to leasable minerals and water power values.

On March 3, 1873 (17 Stat. 607) Congress authorized the sale of coal lands of limited acreage to individuals at $10 to $20 an acre, depending on the distance from a railroad, and in 1907 a new scale of prices was adopted by the Secretary of the Interior, based, more logically, on the quality and thickness of the coal deposits, their depth below the earth's surface, and their accessibility. At that time it was much cheaper and easier to obtain incidental title to coal rights under the homestead laws at $1.25 an acre.

On March 3, 1909, the first *separation act* (35 Stat. 844) became a law. It authorized patents, with a reservation of the coal and mining rights involved to the United States, to persons who in good faith had entered public lands under the nonmineral land laws prior to withdrawal, classification, claim, or report that such lands were valuable for coal. It solved the problem only partially, and the practice thereupon adopted by the department of making coal withdrawals "from all forms of entry" instead of "from coal entry" expedited the more complete solution effected by later acts.

By the act of June 22, 1910 (36 Stat. 583) withdrawn and classified coal lands were declared subject to entry under the homestead, desert land, and Carey acts, provided a waiver of the coal rights accompanied the application, and by the act of April 30, 1912 (37 Stat. 105) the same privilege was extended to State selections and isolated tracts.

The act of August 24, 1912 (37 Stat. 496) extended the separation policy to lands withdrawn for oil and gas in Utah, and finally the act of July 17, 1914 (38 Stat. 509) extended it to all nonmineral filings on public lands theretofore or thereafter

withdrawn in any state for phosphate, nitrate, potash, oil, gas, or asphaltic minerals.

The act of March 4, 1935 (47 Stat. 1470) extended the separation policy to lands withdrawn for sodium and sulphur.

The act of August 1, 1946 (60 Stat. 755) extended the separation policy established by Executive Order 9701, dated March 4, 1946, to all fissionable materials in the disposal and use of all public lands.

Three potash withdrawals, involving 133,829 acres in California and Nevada, were made within a year following passage of the amended 1912 withdrawal act. Later other public lands were withdrawn for potash classification, and a federal potash reserve not now subject to lease was withdrawn east of Carlsbad, N. M., on which the Government has drilled core tests and found a valuable deposit.

Following the withdrawal and classification of public lands for mineral deposits or reservoir and power sites, a system for the dual use of the lands was required, and a mineral leasing system to permit both agricultural and mineral development was widely proposed. The leasing of mineral lands was not new. In 1911 more than 90 pct of the oil production in the United States came from leased fee lands. It was difficult to determine the value of mineral deposits, separate from the surface, particularly the per-acre value of oil lands if outright disposal of the minerals was involved. Instead of a person interested in development being required to advance the entire price of the lands at the outset, a leasing system was proposed whereby payments in the form of royalties would be made as the minerals were produced. A Geological Survey bulletin published in 1913 refers to the uncertainties as to the market value of potash that made the payment of a sales price on an acreage basis undesirable and the payment for the mineral on a royalty basis much more desirable to the producer. Prospecting permits were advocated for exploration of deep-seated deposits and for the safeguarding of prospectors' rights prior to discovery.

Leasing was therefore recommended upon the condition that any leasing law enacted should be framed so as to encourage development, prohibit speculation, and compensate the state in which the lands were located from loss of taxes.

The first mining lease issued in this century on public lands was a coal lease issued in Wyoming under a resolution of Congress passed in 1912.

A review of a limited number of the hearings before the various committees of Congress from 1910 to 1917, on a bill to authorize exploration for, and disposal of, oil, gas, potassium, phosphate, and coal on public lands, reveals that the hearings were concerned largely with oil and gas.

On October 20, 1914 (38 Stat. 743; 48 USC 440) Congress passed an act authorizing the leasing of coal lands in Alaska.

On August 11, 1917 (39 Stat. 462) Congress authorized the Secretary of Agriculture to permit prospecting, development, and utilization of the mineral resources on lands acquired under the act of March 1, 1911 (36 Stat. 961), commonly referred to as the Weeks Act. This act applied primarily to the acquired forest lands in the East. On July 16, 1946, under the President's Reorganization Plan No. 3, mineral leasing and the supervision of prospecting and mining operations of mineral deposits on these and other acquired lands were transferred from the Department of Agriculture to the Department of the Interior. The act of August 7, 1947 (61 Stat. 913) made the Mineral Leasing Act applicable to acquired lands subject to the same conditions as contained in the respective laws on public lands.

Congress, on October 2, 1917, passed an act authorizing the leasing of potash deposits on public lands. On February 25, 1920, the so-called Mineral Leasing Act was passed and became applicable to coal, oil, gas, oil shale, phosphate, and sodium deposits.

Congress, on April 17, 1926 (44 Stat. 301), provided for the leasing of sulphur deposits on public lands in Louisiana, and on July 16, 1932 (47 Stat. 701), amended the act to include New Mexico. One purpose of this act was to alert oil operators to the possibility of finding commercial sulphur deposits.

Practically all the potash consumed in the United States at the turn of the century was imported, principally from Germany. However, in 1910 the availability of this source of supply was threatened by the creation of the German potash cartel and by restrictions imposed by the German government requiring payment of the high cartel prices.

In 1910, W. C. Phalen[3] called attention to the Permian salt deposits in Texas, Oklahoma, Kansas, Colorado, and New Mexico as possible sources of potash salts, and in 1912[4] he published analyses of brines from Searles Lake in California.

On March 4, 1911, Congress appropriated $20,000 to search for a domestic source of mineral potash, which search was continued, with further appropriations, in later years.

The Secretary of the Interior, Franklin K. Lane, before the Committee on Public Lands, in the hearings on HR 14094, 63rd Congress, March 25, 1914, page 14, stated:

> Neither are the placer laws suited to the location of form and areas of potash claims. Where the deposits are in brine, and the method of production is by pumping, for instance, a single well might be utilized for pumping the contents of an entire basin extending far beyond the limits of placer claim. If the deposit is in dry form, the area covered by a single location would be insufficient to justify the expenditure necessary for development of operations.
>
> This bill authorizes the Secretary of the Interior to grant prospecting permits as proposed for oil prospecting to persons who desire to prospect for wet or dry chlorides, sulphates, carbonates, borates, and nitrates of potassium or sodium. The person or corporation to whom such a permit has been issued is to have the exclusive right for not more than three years to prospect and explore for such deposits in an area not to exceed 2,560 acres.

In 1913, 270,720 tons of potash were imported compared with less than 10,000 tons imported annually from 1916 to 1918, inclusive. During World War I the United States was in a serious situation in regard to its potash needs for agriculture, explosives, and other uses. The principal and potential sources were the Searles Lake brines in California, the Bonneville salt flats in western Utah, the alkali lakes in western Nebraska, the solid deposits of leucite in western Wyoming, greensand in New Jersey, alunite in western Colorado and eastern Utah, potash feldspar in several states, sericite slates in Georgia, furnace dust, and organic sources such as kelp from the Pacific Coast, sugar beet and distillery waste, wool scourings, and wood ashes. Domestic production before World War I was confined largely to potash derived from wood ashes. With new sources of potash, principally brines, the domestic supply

increased slowly to 54,800 tons in 1918, but even such an increase in domestic production from 22 states replaced only about one fifth of the 1913 imports. Prices are reported to have reached a high of $10 a unit of K_2O or 50¢ per lb[5] during World War I as compared with 3¢ per lb during prior years and about 2¢ during recent years.

As an emergency war measure, Congress separated potash from the other minerals under consideration for leasing and passed the Potash Leasing Act, of October 2, 1917, which authorized the Secretary of the Interior to issue prospecting permits and leases not to exceed 2,560 acres within any 50-mile square area, restricted the Searles Lake area in California to leasing only, and provided for the leasing of leucite deposits in Wyoming with a reservation to the United States of the undeveloped coal deposits. To stimulate prospecting, a permittee who made a discovery of a valuable deposit was granted the right to a patent of one-fourth the area held under his permit. Under departmental regulations approved March 21, 1918, the remaining lands, if they contained potash in any form specified in the act, were subject to leasing by competitive bidding. However, that provision was modified by the amended regulations, approved March 27, 1926, which gave the permittee a preference right to a lease, provided he applied for a lease at the time he applied for a patent.

The first Federal potash lease of record under the act was issued August 26, 1918, in the Searles Lake area in California, to the West End Consolidated Mining Corp., which is now the West End Chemical Co. However, no potash salts were produced from government-leased lands in California until after the issuance of three leases to the American Potash & Chemical Corp. on October 19, 1939.

Need for the Potash Leasing Act of 1917 and its value in encouraging prospecting for potash are shown by the number of applications for prospecting permits and leases received by the General Land Office, now the Bureau of Land Management. From October 2, 1917, to June 30, 1918, applications were filed for 326 permits and 12 leases; during the fiscal year 1919, 208 applications for permits and 4 for leases; during 1920, 142 applications for permits; and during 1921, 150 applications for permits and 12 for leases. However, many applications for permits and some for leases were denied because of conflicts with prior applications or other reasons.

Among defects in the 1917 act were the awarding of unlimited patents to one-fourth the area embraced in prospecting permits and leasing the remainder of the area. In some cases the land was selected for patent in such a manner as to isolate blocks of government land, precluding or diminishing the possible chances of leasing the remainder of the permit area. To remedy these defects and to unify mineral leasing by harmonizing the Potassium Leasing Act with the Mineral Leasing Act of February 25, 1920 (41 Stat. 437), the 1917 act was superseded by the act of February 7, 1927 (44 Stat. 1057).

The 1927 act continued the minimum royalty of 2 pct on potassium compounds and related products except sodium and provided for the production of associated sodium, magnesium, aluminum, and calcium. A royalty of 12½ pct was established for sodium in the 1920 Mineral Leasing Act. The legislation on sodium, amended on December 11, 1928 (45 Stat. 1019), provided "that nothing in this act shall prohibit the mining and sale of sodium compounds under potassium leases . . . nor the mining and sale of potassium compounds as a byproduct from sodium leases" and reduced the minimum royalty chargeable on sodium compounds to not less than 2 pct at the point of shipment to market.

The Potash Leasing Act of 1927 was further amended by Congress on June 3, 1948: it changed the fixed term from a 20-year lease to a lease for 20 years and so long thereafter as the lessee complies with the terms and conditions of the lease, subject to reasonable adjustment of terms as may be prescribed by the Secretary of the Interior at the end of each 20-year period succeeding the date of the lease. It authorized the Secretary of the Interior to permit suspension of operations when marketing conditions are such that the lease must be operated at a loss.

Applications for potash leases and permits are filed with the manager of the district land office in which the land is located, or, if there is no district land office in the state, with the Bureau of Land Management, Washington, D. C.[5]

Following receipt of an application, the Bureau of Land Management checks the status of the land involved to determine whether any or all of it was open for filing on the date of application. If any of the land is vacant, the application is referred to the U. S. Geological Survey for a report and recommendations. If the land is subject to leasing, terms pertaining to royalty, investment, and minimum production are recommended.

Under the applicable regulations (43 CFR 194) no person, association or corporation may be granted either directly or indirectly an interest in more than 51,200 acres in any one state for prospecting permits and 28,160 acres under lease. The maximum acreage obtainable in one permit or lease is 2,560 acres.

Prospecting permits are issued for a period of 2 years and may be extended for a like period provided an adequate core test has been drilled on the permit land during the initial 2-year period.

Upon making a discovery of a commercial deposit, a permittee is entitled to a preference right lease to any or all the land embraced in the permit, but not to exceed 28,160 acres in any one state. Vacant land known to contain valuable deposits may be offered competitively upon application or on the initiative of the Secretary of the Interior. Royalty rates range from 2.5 pct for sulphates to 5 pct for chlorides, based on the sales value of the crude or refined salts at point of shipment to market. The investment and production requirements are relatively low but sufficient to assure good intention in development of the deposits. The rentals for the first calendar year or fraction thereof are 25¢ an acre, 50¢ an acre for each of the next four calendar years, $1 an acre annually thereafter. The minimum investment on one or more leases may be credited to all leases benefited thereby. Leases are issued for an indefinite period, subject to compliance with the terms of the lease, with a provision for adjustment of terms and compliance with revised regulations and modifications of the leasing act after each 20-year period.

The supervision of prospecting and mining operations, royalty accounting, and the collection of royalties under the potash act are under the jurisdiction of the Mining Branch, Conservation Division, United States Geological Survey, through the Regional Mining Supervisor.[6]

The first core test for potash in the Carlsbad, N. M., area, was drilled on non-Federal land by the E. J. Longyear Exploration Co. in 1917, about 3 miles southeast of Carlsbad. Had Longyear drilled

about 15 miles farther east, a commercial potash deposit might have been discovered at that time rather than 10 years later.

The discovery of potash in the Permian Basin east of Carlsbad was made by an employee of the Snowden-McSweeney Oil Co. who noticed some pink salts in the cuttings from the McNutt No. 1 oil test well about 22 miles northeast of Carlsbad and had them analyzed for potash. When this information came to the attention of the Geological Survey, samples of cuttings were obtained from the company, chemical analyses were made, and the cuttings were examined petrographically. These were the first cuttings from New Mexico to be so examined for potash by the Geological Survey. Previous examinations of cuttings from the Permian Basin had been restricted largely to chemical analyses, but from that time on, in the search for potash, samples collected by the Geological Survey from oil well cuttings and cores were examined mineralogically.

The pink salts in the cuttings from the McNutt No. 1 well were identified in 1925 by R. K. Bailey of the United States Geological Survey as being polyhalite. One sample showed the presence of sylvite which analyzed the equivalent of 3.09 pct K_2O. When the Survey's report was received by V. H. McNutt, geologist for Snowden-McSweeney, he recognized the significance of the value of pure sylvite with 63.2 pct K_2O, in comparison with polyhalite with 15.6 pct K_2O. On application by McNutt, a prospecting permit was issued in 1926, and the first core test on a potash permit in New Mexico was drilled, resulting in the discovery of deposits of polyhalite, sylvite, carnallite, and langbeinite. Although the grade of potash salts was relatively low, considerable interest was created and the Snowden-McSweeney Co., the operator, decided to continue prospecting in the hope of finding a higher grade ore that could be economically developed. However, not until its fourteenth and sixteenth holes were drilled did the company find what it considered a potentially commercial deposit.

The American Potash Co., subsequently the United States Potash Co., was organized to develop the deposit and on March 1, 1931, the first commercial shipment of run-of-mine salts was made. The first shipment of refined salts was made in 1932.

In 1931, following the initial production, a second group, later incorporated as the Potash Co. of America, found another promising area. Development was begun, and the first shipment of run-of-mine salts was made on February 20, 1934. The Potash Co. of America obtained a patent on its flotation method for the separation of sylvite and halite and constructed a plant.

Foreign producers of potash, realizing the effect of these competing sources for the American market and knowing the high freight rate from Carlsbad to eastern markets as compared with ocean freight rates from Europe, cut prices in the hope of regaining American markets. To permit the American producers to become established on a firm financial basis to compete with foreign producers and to assure a domestic supply of potash particularly during emergencies, the Secretary of the Interior suspended the issuance of prospecting permits for about 10 years, from 1934 to 1945. During this period, a third group of permittees, who had obtained permits prior to the suspension order, found a commercial orebody that was developed by what is now the International Minerals & Chemical Corp.

Duval Sulphur & Potash Co. and Southwest Potash Co. also have begun commercial operations.

Summary

In 1920 the production and sales of potash in the United States by the 66 plants reporting in terms of K_2O were 41,444 tons valued at $7,463,026 and imports for fertilizer use were 197,795 tons valued at $33,885,627, making a total of 239,239 tons valued at $41,348,653. In 1950 the production and sales were 1,275,494 tons valued at $39,695,038 and imports were 192,884 tons valued at $11,771,958, making a total of 1,468,378 tons valued at $51,466,996. The difference in value per ton between domestic production and imports is due largely to the point of valuation for domestic production being the plant and for imports the eastern ports of the United States. The delivery of 114,279 tons to Illinois for agriculture exceeded that to any other state.

According to the Geological Survey's records as of June 30, 1952, 1306 potash prospecting permits have been issued in 13 states and 52 potash leases in 3 states. On that date, there were in effect 174 prospecting permits in 5 states and 33 leases in 2 states.

Royalties and rentals payable during the fiscal year 1952 under the provisions of the Potash Leasing Act amounted to $2,016,575.

In carrying out recommendations made in the early hearings on the mineral leasing acts, Congress provided that 37½ pct of all moneys received for bonuses, rentals, and royalties be paid to the respective states in which the leases were located to be used for public schools and roads, and that 52½ pct be paid into the General Reclamation Fund. The United States Treasury retains only 10 pct.

Only a small portion of the 3000 square miles in southeastern New Mexico in which potash has been found in oil-well cuttings has been prospected by core drilling.

Potash had been found in a large area of the Paradox Basin in southeastern Utah and southwestern Colorado.[8]

It is not known whether the potash deposits found in Saskatchewan, Canada, extend into Montana and North Dakota. Examination of well cuttings and brines and radioactive logging of the wells drilled for oil and gas in those states may reveal this.

It is not anticipated that production from the brines of Searles Lake in California or western Utah will be greatly increased.

The outlook for the future indicates that production in the United States in 1955 will be double that of 1950, the last year for which complete information is available. About 90 pct of the present production comes from New Mexico and about 10 pct from other states. Potential reserves of soluble potash salts appear adequate for many years to come.

Selected Bibliography

[1] George Otis Smith et al.: Classification of the Public Lands, U.S.G.S. *Bull. 537* (1913) pp. 35, 48, 49.

[2] Potash Salts, Advance Chapter Mineral Resources of the United States, U.S.G.S. (1910) p. 23.

[3] Potash Salts, Advance Chapter Mineral Resources of the United States, U.S.G.S. (1912) p. 15.

[4] C. A. Brown, Assistant Chief, Bureau of Chemistry and Soils, U. S. Department of Agriculture: *The Federal Review* (June 1925).

[5] Code of Federal Regulations, Title 43, Ch. 1, Part 194.

[6] Code of Federal Regulations, Title 30, Part 231.

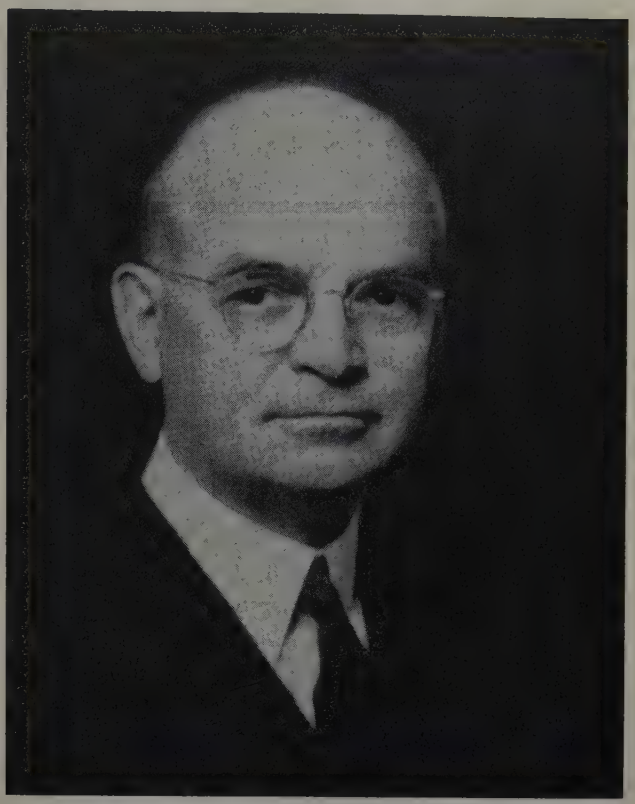
[7] H. I. Smith: Potash, Ch. 34, *Industrial Minerals and Rocks*, AIME 1949, p. 699.

[8] B. W. Dyer: Discoveries of Potash in Eastern Utah. *Mining Technology* AIME (January 1945).

The Daniel C. Jackling Lecture

The Award —

An annual invitational address by an outstanding man in mining, geology, or geophysics who has contributed significantly to the progress of technology in these fields.



First Lecturer —

RENO H. SALES

The Presentation —

by Fred Searls, Jr.

Our colleagues cannot in the future earn reputations and medals for achievements in milling and smelting ore and for successful management of mining companies, if some one doesn't keep finding and mining the ore to mill and smelt and to provide the basis for the good management. Good ore seems sometimes to be quite helpful to good management, and good ore is not as easy to find as it used to be. And there are still *more* reasons, why—when this honor had been decided upon and named for Mr. Daniel C. Jackling—it should be initially conferred on Reno H. Sales.

The terms of reference of the award are that it should be conferred upon an individual working in the fields of mining, geology, geophysics, who has made an outstanding contribution to the progress of technology in one or more of those fields. And in this initial year (the earlier years are the easiest) just that was done. I think I could get away with it amongst the old-timers of my generation if I say that this time they made the award to *the* individual, who made *the* outstanding contribution. But, as Al Smith used to say, "Let's look at the record."

Really, the record is mixed up a little with extra-lateral litigation—the old-time "apex cases" under Section 2322 of the Revised Statutes. Reno Sales doesn't like such litigation, but there is little question that the urgency, generated by conflicting claims of intra-limital and extra-lateral rights, and consequent necessity for committing to records and maps the complications of Butte structure, did advance—and by several years—the first continuous, accurate, systematic, and scientific portrayal of the geological and mineralogical details of what is, after all, the most important and the most complicated copper district—at least in North America.

Prior to Sales' time, some of the most eminent of geologists often waived details. One geologist took the judge to the window to show him the Rarus fault going through a notch in the Continental Divide. The entertaining testimony in the Bluebird litigation was as perfect in literary style as it was innocent of the criteria by which faulting and lack of continuity of vein structure are now recognized.

At any rate, this need for an accurate record of structure was in the background—to whatever extent it was, or was not, the etiological agent—for formation of the Geological Department of the Amalgamated Copper Co. in and about the year 1900. Charles W. Goodale was at that time the head of the Boston & Montana, and John D. Ryan succeeded William Scallon as the president of Anaconda Copper Mining Co. in 1905. Horace V. Winchell, for years a specialist in extra-lateral litigation, was perhaps the man who at that date had the vision, the persuasive weight with the management, and the willingness to defend the idea of large current expenditures (as then viewed) for a complete and sys-

This page of Mining Transactions AIME follows p. 424. The intervening non-Transactions pages appeared in Mining Engineering.

tematic Geological Department, for which there was no real precedent in the industry. Brunton, consulting engineer for the Amalgamated, backed him up. At that, the department started to function with one geologist, one assistant geologist, and a draftsman. In 1913 there were about 11 geologists, in 1928 15 geologists, 44 engineers, and 67 samplers.

But Sales, who took over from Winchell in 1906, is the man who made it tick. He graduated from Montana State College at his home town in Bozeman (in 1898) but although born in Iowa went on to Columbia, where he received an E. M. degree in 1900. He immediately went to work for the Boston & Montana under Mr. Goodale. By 1913, Sales published his *Ore Deposits at Butte, Montana,* which was eagerly adopted by the Clark and North and East Butte operators as a great advance over Weed's Professional Paper, which—though published in 1912—represented work done before 1906. The Anaconda Geological Dept. was very soon accepted in every camp in the country as a model whose methods were to be used to as full an extent as the production of smaller districts warranted. Sales said and wrote as early as 1927, "The viewpoint of the mining geologist and his objective is to find all of the ore in a district at a minimum of development and mining cost."

One of the reasons his methods spread so rapidly is that they were early put at the disposal of the leading educators in geology, again through the employment of the latter as professional witnesses in Butte litigation. He instructed the instructors. Leading authorities from universities studied Anaconda maps, took Anaconda notebooks underground, made notes as nearly like Reno Sales' notes as they could, and returned to their classes to spread the method, until now there is no other, and it takes someone like me to realize that there was ever any other way. It wouldn't be right to name names, but I have waited while an eminent and highly respected professor of geology occupied the time of the Court, and of a suite of highly-paid lawyers and experts, for twenty-two minutes by the clock while he hunted through 175 pages of handwritten underground notes in order to answer a question in cross-examination. The art of study of the structure of developed ore deposits belongs to two periods—one pre-Reno Sales, and the other post-Reno Sales—and the second will endure.

Nor is this his first honorary award. He received the Penrose Medal from the Society of Economic Geologists in 1939 and the Egleston Medal from Columbia in 1942.

That isn't all, but the rest is so new that you all know all about it. Reno retired as active head of Anaconda's Geology Dept. Jan. 1, 1948. He has never been very patient with poor observation or faulty geological reasoning. When Reno corrected some earlier work of others in his 1948 paper with Meyer, Hulin commented: "The authors are certainly to be congratulated, and the presentation and publication of this paper unquestionably marks a tremendous step forward in our understanding and knowledge of wall rock alteration." And Johnson: "This paper did not come from a university; it did not come from a governmental institution; it came from a mining company."

Sales has added more, not only to our knowledge of ore deposits, but to the attitude and methods for their successful study, than any man of any age. Usually, prior to Sales' time eminent geologists, even in this country, and many of the early members of our own Survey—and I except a few like G. K. Gilbert—after some observation, consideration, and thought, announced theories first and adduced certain evidence to confirm their ideas. But with Sales' department came the idea, the methods, and the insistence on a new technique. "Let's look at it. Put it all down correctly on a piece of paper, including details. What are the facts? How do you explain this feature and that one, etc.?" It is, I think, not too much to say that the approach revolutionized the economic mining geology of 40 years ago. Until recently mining geology has been largely structure, faults, zoning and rock alteration.

From the ranks of the young geologists, and also from the ranks of the geophysicists, will be drawn future Jackling Award Lecturers. A few of them are very, very sharp, but I believe they can draw from this, our First Lecturer, much to their advantage, if they will absorb his passion for careful and meticulous observation, and his patience in waiting to announce a conclusion until the evidence is in and carefully weighed.

The Acceptance —

by Reno H. Sales

I deeply appreciate this high honor, coming from a great organization whose written history will be a record of great achievements and progress in the arts of mining and metallurgy, not only in this country but throughout the world. I can say only that from my first days in the mines at Butte up to now my goal has been to do all within my capabilities to make geology useful and helpful to the prospector, to the miner, and the operator, to the end that more deposits of useful metals be found and that the maximum of metal recovery be made from mines and districts in the process of development.

I am proud of the name of Jackling on the medal because the name D. C. Jackling will live always in the history of copper mining. How well he deserves his title "the father of the porphyry coppers."

I shall treasure the medal, and carry with me always a deep feeling of gratitude toward this worldwide organization and its membership for doing me this very great honor.—I thank you very much.

1954 Award Committee

E. P. Pfleider E. D. Gardner LeRoy Scharon J. D. Forrester Guy Bjorge Harrison Schmitt Herbert Hoover, Jr.

The First Jackling Lecture

Genetic Relations Between Granites, Porphyries, And Associated Copper Deposits

by Reno H. Sales

EVERY mine manager, mine geologist, and every prospector in the field who appraises the future of mining properties does so on the basis of a theory of ore deposition whether he recognizes this fact or not. Wherever the ultimate value of a mine or prospect is dependent upon undeveloped ore, any estimate by an engineer or geologist necessarily involves a theory of origin of the deposit. In view of these facts the importance of a thorough understanding of the genesis of ore deposits cannot be overestimated. It well may be that the greatness of our country will depend upon finding reserves of natural resources as yet undiscovered.

The genesis of ore deposits offers a fascinating field for study and investigation. This is attested by the large number of geologists who have written at length on the subject. For progress made toward a better understanding of ore genesis we owe much to Kemp, Lindgren, Ransome, Vogt, Emmons, Spurr, Graton, and a host of others. Within my own experience, I have attempted to apply in practice the various concepts and theories that have been advanced by these men.

Because of the fact that most nonferrous metal deposits are related to igneous activity, many leading authorities, notably Bowen, Fenner, and Ross, have contributed greatly to our knowledge of the origin of ores. These petrologists postulate that through processes of fractional crystallization, a granite magma differentiates into successively more acid phases from which, through further crystallization, the solutions responsible for ores finally emerge. It is stated that these solutions find their way into fissures in the solid hood, then ascend to near surface zones where they deposit their mineral load. This appears logical and sound, but as applied to the copper deposits to be considered here today, the vital role played by the so-called *porphyry* intrusions as an intermediate step in the process seems to have been overlooked.

The author believes all geologists will agree that theories involving ore solutions and their origin should be based as far as possible upon available facts. Unfortunately, the facts at source points are not disclosed for observation. The next best procedure, then, is to project known information to the assumed source, but any theory so evolved not in full accord with observed field relations should not be accepted as final. Therefore, in our search for answers to problems involving the nature and origin of ore-forming solutions, we must examine not only the structure and mineral composition of the ores and the rocks enclosing them, but also wall rock alteration effects and any other factual evidence available bearing on the problem. However, we must never lose sight of the fact that the vertical range of mineral deposition open for examination and study in most deposits may be small by comparison with the distance from the solution source to the zone accessible to the observer. This is unquestionably true with respect to most hydrothermal vein deposits of copper.

Mineral zoning is a recognizable feature common to many mineral deposits, especially of the fissure vein type. Zoning may be present but obscure where complex structural conditions prevail, or where there have been successive periods of fissuring and mineralization. Obviously, conclusions as to the nature and composition of hydrothermal ore solutions at the point of origin, based upon observation of a limited vertical range in the whole mineral depositional column from source to orebody, are subject to error. To illustrate, at Butte nearly 5000 ft of vertical mine development has exposed a clear mineral zoning picture. If underground mine development up to the present time had been confined to the first 1800 ft of depth in the so-called peripheral zone, the vein minerals exposed would have included quartz, rhodochrosite, rhodonite, and sphalerite, with subordinate galena and pyrite, all in association with extensively argillized wallrock containing calcite veinlets, with very minor sericite

R. H. SALES, Member AIME, is Consulting Geologist, Anaconda Copper Mining Co., Butte, Mont.

Discussion on this paper, TP 37861, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Feb. 16, 1954. New York Meeting, February 1954.

along vein boundaries. It is difficult to believe that a theory of the nature of the ore solution at its source, based upon the facts disclosed in the 1800 ft of vertical range, would have been the same as is now postulated after taking into account also the facts disclosed at deeper levels where wide zones of sericitic alteration enclose rich copper ores which consist of chalcocite, enargite, bornite, pyrite and quartz.

Earlier literature on ore deposits, in the main, supported the theory that successive zones of sericitic and argillic alteration are products of separate solution surges of different chemical composition and that sericitic alteration is solely a product of alkaline solutions. However, it is believed that at Butte the ore solutions arriving at deepest points now open to observation were probably acid and that during their upward journey they produced, simultaneously, successive bands of sericite and argillic clays in the wall rock outward from the fissures. Throughout the observable vertical range of ore deposition, the alteration pattern is the same qualitatively, but quantitatively, sericite greatly predominates over clays at levels nearest the solution source, while the reverse is true at greater distances from the source. Here again, as with vein mineral composition, Butte illustrates clearly that errors may creep in if the nature of a primary ore solution at the point of origin is judged from wall rock alteration effects at a single point of reference, or from any zone of limited vertical range far distant from the solution source.

The problem of the origin and nature of the ore solutions responsible for copper deposits genetically related to granites and intrusive porphyries is, as already has been stated, far from simple. All are of magmatic origin, but in means and methods of transfer of their mineral content from the depths wholly different characteristics may be exhibited; therefore generalized statements are subject to error. In sharp contrast, where generalizations may be made with confidence, the copper deposits of Butte and the Magma copper mine at Superior, Ariz., are good examples. Both are of hydrothermal origin associated with quartz-porphyry dikes. In mineral content they are the same, qualitatively, consisting of chalcopyrite, bornite, enargite, chalcocite, sphalerite, rhodochrosite, quartz and pyrite. The ore fluids, containing in abundance copper, zinc, arsenic and manganese, with silica, iron and a high sulphur content, effected the same type of wall-rock alteration. Thus, based on mineralogy and wall-rock alteration the ore solutions at Magma and Butte are considered to have been similar in character and composition. As to source, both followed intrusions of quartz-porphyry. At Butte, granite encloses the orebodies. At Magma, underlying granite may be inferred with good reason because of outcropping granites at short distances to the north and west.

But the fact must not be ignored that even with ore solutions of similar chemical composition at the source, the resulting ore deposits may differ widely in physical or mineralogical character because of the influence of wallrocks of varying chemical composition, or because of the nature and availability of channelways for solution travel. Where ore solution channels intersect limestone formations, massive sulphide replacements are the usual result, as at Bisbee, Bingham, and Morenci, for example. The Butte veins are typical of fissure vein deposits in granite or similar igneous rocks of relatively uniform chemical composition. It sometimes happens, however, that there has been no well-defined premineral fissuring of the rock formation containing the orebodies at or directly above the solution source. Under such a condition, which prevails over the Cananea district in Mexico, breccia-pipe structures are formed by solutions or gases acting under high pressure, and these breccia-pipe structures present the only avenues of escape for the ore solutions derived from deeper magmatic sources. And before making direct comparisons of districts we should ask ourselves this question: Which segment in the vertical range of solution activity between the magmatic source and the outer limits of the ore deposit has been made accessible to us by erosion? For example, had there been less erosion, Morenci might not have been developed as porphyry copper but only as a district of replacement orebodies in sedimentary rocks.

Examples for consideration here are certain types of copper deposits known as porphyry coppers, and their near relatives, Butte and Chuquicamata. As already indicated these are of igneous origin and directly associated with what are commonly designated quartz-porphyries or monzonite-porphyries. The economic importance of this class of deposits may be judged by the fact that during the past twenty years they were the source of more than 85 pct of the copper production of the United States. For the purpose of this analysis it will be convenient to divide these important copper occurrences into three general types: 1—magmatic, of which the Colorada orebody at Cananea is the outstanding example; 2—late-magmatic, represented by the Ajo and Potrerillos deposits; and 3—hydrothermal, of which type Butte is the classic example.

Magmatic Type: The Colorada orebody at Cananea, Mexico, is intimately associated with quartz-porphyry intruding andesitic rocks. The orebody is very high grade, having produced over 750 million lb of copper. The primary ore is composed of massive glassy quartz, phlogopite mica, chalcopyrite, bornite, molybdenite and pyrite, with minor covellite and chalcocite. There is no orthoclase associated with the ore minerals nor is there any evidence of a post-porphyry introduction of orthoclase into the rocks enclosing the ore.

It appears beyond question that the mineral content of this "cup-shaped" deposit existed as a fluid ore-magma mass at its present position and there underwent a process of segregation, quartz being the first to crystallize out in solid form, followed by mica, copper sulphides, molybdenite and minor quartz. The enclosing wall rocks were altered locally, and a thin halo of copper enrichment of the wall rock was effected by injected dike-like veinlets of quartz and chalcopyrite which were supplied by the ore fluid.

The mechanics of the process of transfer of the ore from a deeper source to its present position is a debatable question. Possibly it could have been brought up en masse with the intruding porphyry, but more likely it was injected from below as an ore magma during the final crystallizing stages of the deeper portions of the porphyry. Apparently it was trapped at the approximate position where the nearly vertical cylindrical body of porphyry, in its upward extension toward the surface, divides into a number of prongs or dikes.

Near Chuquicamata, Chile, there are four small

pegmatitic copper ore deposits consisting of coarse glassy quartz, orthoclase, mica, chalcopyrite, bornite and minor molybdenite. They are isolated occurrences in irregular dike-like bodies of granite-porphyry intruding granite. They show no genetic connection with fissures or brecciation. Just as in the case of the Colorada deposit, the facts of geology involved do not support a theory of introduction of the ores by hydrothermal solutions.

Late-Magmatic Type. Ajo and Potrerillos: The copper deposits of Ajo, Ariz., and Potrerillos, Chile, are of the late-magmatic type, and no doubt there are others. The author might designate these deposits more accurately as combination magmatic-hydrothermal, because both kinds of mineral deposition are present in each. They exhibit 1—an intrusion of what might be called a "porphyry" ore magma, and 2—a later addition of hydrothermal mineralization. They differ from the Morenci hydrothermal type of porphyry-copper inasmuch as in these two examples the porphyry-magma intrusion appears to have brought up with it a part of the copper which was later to be distributed during cooling and crystallization as disseminations, seams and veinlets of chalcopyrite and bornite throughout portions of the porphyry mass.

The intruded porphyry bodies are projections from deeper granite magmas, expelled at a time when higher portions of the magmas, possibly developing cupolas, had reached an intermediate stage in the processes of differentiation. Neither at Ajo nor at Potrerillos did these intruded fractions of the granite magma reach surface; they came to rest under a heavy covering, mostly volcanics at Ajo and a limestone series at Potrerillos. Both porphyry masses are roughly bulbous in form exhibiting greatly expanded upper parts but contracting downward to narrower bodies extending in depth no doubt to the parent granitic magmas.

Within these cooling masses of intruded magma, the greater portions of which have been eroded, mineralization from the crystallizing parent magma continued up to the time the porphyry masses were completely solidified. Geochemical processes through which the respective copper orebodies developed to the final stages were identical in the two deposits but less obscure at Potrerillos than at Ajo. At Potrerillos the primary copper minerals, chalcopyrite and bornite, with associated orthoclase and veinlets of quartz, are distributed throughout the porphyry but not uniformly. Limited portions are minable as ore. The quartz seams apparently represent the residual excess silica of the crystallizing magma, but they are not accompanied by any alteration of feldspar grains with which they are in contact. The introduced orthoclase, also of magmatic derivation, is pre-sulphide in time of crystallization and shows no alteration where in contact with the copper sulphides. There is relatively little pyrite, indicating that the sulphur content of the magma residue at the time of the porphyry intrusions was very small.

The late hydrothermal mineral additions to the easterly fringe of the Ajo orebody were derived from a late stage in the processes of differentiation in the underlying parent granitic magma, after sulphur had become an important constituent of the magmatic ore solution. At Potrerillos, the fissures acting as passageways for the late high-sulphur solutions depositing ore minerals, including enargite, covellite, pyrite and quartz, are well marked structural features, but at Ajo the fissure pattern through which hydrothermal solutions were introduced is not well defined.

An alternate to the theory of origin above set out may be suggested here, which is that the intrusion and final solidification of the porphyry was too early in the process of differentiation taking place in the granite magma to have brought with it more than insignificant quantities of copper. Under this premise, a fissure tapping the magma residue at a later but pre-hydrothermal stage of differentiation could have brought in the orthoclase-copper mineralization of the ore zone. Such an ore solution, low in sulphur, would probably have been alkaline in reaction. One objection to this hypothesis is the absence of fissuring at Ajo of the strength and type that would persist as ore channels to a deeper solution source. Furthermore, without some kind of a solution stoppage or cut-off effecting a time lapse, it seems difficult to explain the change from the orthoclase-copper mineral deposition requiring an alkaline feldspar-stable environment, to a high sulphur, hydrothermal type of mineralizing solution.

Hydrothermal Type: Butte has been cited as the classic example of this type. Among other well-known copper deposits belonging in this class are Morenci, Chino, Ely, Miami-Inspiration, Bingham, Bisbee, Magma, Cananea, and Chuquicamata. The members of this group of hydrothermal deposits resulted from definite orderly processes taking place in a crystallizing granite magma. This sequence of events in chronological order was:

1—Invasion of a granitic type of batholith.

2—Cooling and crystallization of the granite magma accompanied by processes of differentiation and "fractional crystallization."

3—Intrusion of a portion of the upper layer of the liquid granite magma into the already solid granite hood, and in some cases into the roof rocks.

4—Crustal fissuring tapping the magmatic solutions at their source.

5—Ore solutions ascending through fissures or other openings to deposit ores.

The copper deposits developed through the above series of events differ widely in their structural and geologic setting, as indeed they should in view of the variables involved, including composition and volume of the parent magma, its original metal content, and the numerous factors mentioned earlier. All of these variables shared to some extent in the final determination of the physical and mineralogical character of the deposit. It would be a miracle if two were found exactly alike. The remarkable thing about it is that they have enough features in common to enable us, with some confidence, to draw conclusions as to their origin.

It has already been noted that a single deposit, Potrerillos for example, may exhibit two types of mineral deposition, one involving a feldspar stable solution, the other a high sulphur hydrothermal solution; the former probably alkaline in reaction, the latter neutral or acid. The late-magmatic or *orthoclase* depositional phase is believed to be a near source phenomenon, in marked contrast to the far-from-source deposition of ore from hydrothermal solutions.

A logical conclusion is that one could expect to find a deposit of either type or a combination of the two. Chuquicamata, for example, shows both late-magmatic and hydrothermal phases of mineral deposition, but there the hydrothermal ores greatly

predominate, which is in reverse order, quantitatively, to that at Potrerillos.

Because of the fact, as proved at Butte, that the intense high-sulphur hydrothermal solution attack converts feldspars, including orthoclase, to sericite, the question is raised as to whether or not a late-magmatic orthoclase type of mineral deposition could have been present in certain copper deposits where we now see only evidence of intense hydrothermal activity in connection with the deposition of sulphide ores. Whether the hydrothermal solutions that eliminated the feldspars also effected a solution and re-deposition of an early copper sulphide minerals poses an interesting question.

At Butte, and most other hydrothermal-type copper deposits being considered, the intruding porphyry magma does not bring with it more than small traces of the metals of the ores; therefore it is required that the intrusion create a condition at its source favorable to the development and concentration of magmatic ore solutions which later ascend through fissures to form orebodies.

With reference to the problem of genesis, Butte has been selected for a more detailed analysis because of the extensive range of vertical and lateral mine developments open for examination and study, and because of the author's familiarity with geological conditions there. It will be impossible in the time allowed to describe in detail the other named deposits, or to make more than brief mention of the many interesting facts having to do with the genesis of the Butte ores. It should be noted here that my descriptions and considerations have reference only to primary or hypogene mineralization.

Butte is an area exhibiting extremely complex fissure systems of different ages, of which the two earliest, known as the East-West and Blue vein systems, are heavily mineralized, having produced very large tonnages of high-grade ores of copper, zinc and manganese. The deposition of copper ores consisting of chalcocite, enargite, bornite and chalcopyrite was accompanied by intense sericitic alteration of the wallrock. The ores of copper, zinc and manganese are zonally distributed in the order named, outward and upward from zones of highest alteration intensity. Four quartz-porphyry dikes of pre-vein age traverse the central and eastern portions of the district which coincide, significantly, with the largest area of intense alteration.

Brief descriptions of the quartz-porphyry dikes, alteration zones, and composition of the mineralizing solutions seem necessary here as a preliminary to the consideration of the problem of ore genesis, because these are the only observable facts that throw light upon the nature, origin and composition of the ore solution at its source.

The quartz-porphyry occurs in dike form within the vertical range exposed for observation, with one exception. In the East Colusa mine area, the Modoc dike is changing to an irregular stock-like form in the deep mine levels. In east-west distribution, the area of most numerous dikes extends from the Mountain View to the Pittsmont mine area. This area may be regarded as the center of porphyry activity marking the approximate position of the underlying source magma, a geological feature to be given emphasis later. It is also true, district-wise, that the cross-sectional area of quartz-porphyry is increasing with depth. A structural relation of some significance between the porphyry dikes, pre-porphyry fissures, and the earliest veins should be noted. The structure patterns of the dikes and the early East-West veins are similar in the area of maximum porphyry activity. Both strike southeasterly and dip southerly in the Mt. View and Berkeley mines. Easterly from that area, the dikes and veins change to northeasterly strikes and northerly dips, indicating that the same stresses were operative before and after the intrusion of the porphyry. These pre-porphyry fissures may have been the trigger setting off the series of events culminating in the formation of the Butte deposits.

Alteration

Butte orebodies were formed chiefly by metasomatic replacement of wall rocks. In order that the original fissures or the veins under construction could have continued to act as passageways for solution travel during the growth of the veins, it is required that the attacking solution dissolve and carry away replaced rock materials more rapidly by volume than the replacing vein minerals are being deposited. Otherwise, the conduit would become choked or dammed and solution travel stopped.

At all times during mineralizing periods, the sericite fronts were migrating outward from solution channelways with the highest rates of advance at deeper horizons, in conformity with the maximum of intensity and potency of the ore-bearing solutions. The horizontal cross-sectional area of sericitization, greatest at deep levels, was increasing at successively higher points of reference in vertical range as long as hydrothermal activity continued. In the case of the Central zone, sericitization of the granite between earliest veins was completed upward to and above the present erosion surface within fairly well-defined lateral limits. In surrounding areas, however, it was only within the veins and in their immediately adjacent wallrocks that sericitization extended as high as the present surface, the intervening granite at high levels being fresh or only slightly altered.

In the broad picture of alteration at Butte in deep mine levels outside the Central zone, sericitic alteration is most widespread where fissures are closely spaced. Upward from any of these outside localized areas where alteration is extensive, the change to fresh granite between veins is not sudden but gradual, and it is characterized by argillic alteration along joint planes, cracks, and weak shear zones, all with gradually decreasing intensity upward from the more highly altered area. In vertical thickness this zone of gradual change is extremely irregular. District-wise, then, wallrock alteration in all its phases of intensity must be viewed as having been migratory upward from deep horizons from the beginning.

Composition and Origin of the Ore Solution

Upon the basis of minerals added to the fissures and wall rocks, the ascending ore solutions arriving at horizons of deepest mine levels must have contained principally sulphur, copper, zinc, lead, manganese, silica, arsenic, carbon dioxide and iron, with minor fluorine and boron. They carried also any alkali or base radicals leached from the wallrocks at lower horizons. Judging from alteration effects they were probably acid in reaction. In what chemical combinations these elements may have been held during upward travel is indeterminate.

The series of events occurring at Butte were: 1—cooling and crystallization of the quartz-monzonite magma, with concurrent development of

aplite and pegmatite dikes; 2—intrusion of "quartz-porphyry" dikes cutting the granite, aplite, and pegmatite dikes; 3—crustal fissuring of the granite and porphyry; and 4—ore solutions rising through the fissures from a deep magmatic source and depositing the ores.

The quartz-porphyry was intruded before the solid granite hood had reached a great thickness, and before the processes of differentiation and fractional crystallization had gone on long enough to effect much change in the composition of the granite magma beneath the solid hood. It is here postulated that the intruded porphyry was not expelled from a distinct and separate quartz-porphyry magma but instead was an injection of a portion of the granite magma. A porphyritic texture developed at near-surface horizons where we now see it owing to its having been thrust as dikes into a relatively cool crust. Significantly, in the East Colusa mine, as previously noted, the horizontal cross-sectional area of the porphyry is increasing rapidly with depth and changing in form from narrow dikes to a stock-like body that is irregular in outline.

The vitally important deduction made from a study of the behavior of the porphyry dikes of the Butte area, and applicable to other copper deposits related to porphyries, is that the upward invasion of a portion of the granite magma brought about the development of a *sub-hood* cupola projecting above the main body of the magma into the solid hood of the granite itself. This upward projecting liquid mass became a closed system at the moment the narrow dike extensions solidified in the upper cooler part of the granite hood. The lower cupola mass was encased in the lowest layers of the solid granite hood having temperatures not greatly below that of the underlying magma, and because of this fact, the magma occupying the cupola remained fluid as the zone of magma solidification, including the top and walls of the cupola, migrated downward. This cupola and its magma environment presented a very favorable locus for the collection and concentration of water, sulphur, and the metals of the ores, a theory advocated by B. S. Butler and W. H. Emmons for granite cupolas extending into roof rocks.

The effect of the porphyry dike intrusion at Butte was to modify internal cooling stresses within the solid hood of granite and to cause thinning and weakening of the granite above the cupola, thus preparing it for the later initiation and development of fractures of the early East-West vein system.

Through the processes of differentiation and fractional crystallization which continued in the granite magma beneath the cupola hood, there was eventually developed what has been referred to as an *aqueous magmatic residue,* by others called the *magma residuum.* In early stages it was composed of water, volatiles, with probably some sulphur and metals, and the materials which form orthoclase and mica. But in later stages with the feldspars and micas crystallized out, the residue became the ore solution consisting of water, silica, sulphur, carbonic acid, and the metals of the veins and ores, later tapped by East-West fissuring to begin the formation of orebodies in higher levels. As long as orthoclase was crystallizing out, the magmatic residue was undoubtedly alkalic in character and reaction. But at the same time the alkalies were crystallizing out forming feldspars, the magmatic residue was being further enriched in sulphur, water, chlorine, carbonic acid, and other acid-forming constituents, none of which combines with other elements to form rock minerals of the monzonite. It is reasonable to assume, therefore, that in a closed system with a decreasing ratio of alkalies to acid constituents the "residue" would eventually become neutral or possibly acid in character, and remain so unless and until something occurred to cause it to revert to alkalinity. In this situation the magma is supplying the alkali radicals to the crystal mesh at a more or less constant rate, but there is simultaneous loss of these alkalies because of their crystallization to form orthoclase.

It follows that the composition and nature of an intrusion of a portion of the magma, or of an ore solution tapped from an aqueous magma, would depend upon the timing of the fracturing in relation to the stage of the "residue" development. At Butte the East-West fissures did not appear until relatively late in the process when the solutions were rich in water and sulphur, together with copper, arsenic, and other metallic elements now present in the Butte veins. It is believed that this late magmatic residue, or ore solution, was being held largely in what Gilluly terms "the crystal mesh", a zone of crystallizing magma topped by the hood and supported below by liquid magma. This in effect means that the "ore solution" was in part interstitial material dispersed generally in a large volume of formed or partly formed crystals.

The underground observed facts in Butte support this idea inasmuch as the period required for the build-up of the large orebodies common to the early East-West veins and their accompanying zones of alteration must have been a relatively long one. There can be little doubt, however, that the rate of upward flow of the ore fluids in the tapping fissures was affected adversely by the hydrostatic head and frictional resistance against which they had to move. Because of this it is not improbable that if the ore carrier was a liquid at the source before and at the moment of release, and at a temperature below the critical point, it would remain liquid after release into fissures. And it is also a reasonable deduction that in its journey upward against continuing pressure and decreasing temperature, a balance would be maintained keeping the ore fluid to and through the zone of ore deposition. This possibility was expressed by Ross in 1928:

> If it (the solution) escapes at high temperatures to a region of low pressure it will undoubtedly change to a gas phase, and its further reactions will be of the gas phase type. If, on the other hand, it is under sufficient pressure and escapes slowly through sufficiently tight channels, pressure may be maintained, and the water-rich, vein-forming materials may remain in the liquid phase throughout their mineralizing career.

Mineralization Periods

The successive periods of mineralization at Butte present an interesting problem. The question arises: What are the underlying causes? Are they due merely to a succession of time-spaced fractures tapping a continuous flow of "magma ore solution" from its place of origin? Or, was there an actual cessation of mineral solution flow over a substantial period of time, afterward rejuvenated by the tapping of a continuing accumulation of solutions by fissures definitely later in age than the earliest known fis-

sures of the East-West system? The following facts have a bearing on these questions.

At all levels the East-West veins are vuggy and they are enclosed in altered rock. When first opened by mine workings a heavy flow of water shows both the veins and altered rock to be excellent water channels. This fact demonstrates beyond question that ore solution travel through these early veins did not stop because of plugging. The cause must have been deep-seated.

A time lapse between the close of East-West mineralization activity and the beginning of Blue Vein ore deposition is indicated by differences in mineral composition of the two vein systems. The early veins are very quartzy, with high proportions of zinc, manganese and enargite, in strong contrast to the ores of the Blue veins which are low in zinc and manganese, with much less enargite in proportion to chalcocite and bornite.

There is always the question as to how much of the mineralization of the earlier veins was added during the Blue Vein mineralizing activity. There is abundant proof at intersections in higher levels that the East-West veins had grown to their full widths, and they show no enrichment at Blue Vein intersections. However, at deeper levels enrichment of East-West veins at crossings by Blue fractures is common, which is as it should be because the vertical range of mineral deposition had moved considerably lower prior to and during the span of Blue Vein activity.

The tops or upper limits of Blue Vein copper ore shoots are universally at lower elevations than those of the East-West veins by as much as 500 to 1500 ft. Such a marked difference would have been unlikely under a condition of sustained flow of mineralizing solutions from a common source. This difference in upper limits of mineral deposition in the two vein systems is indicative of a downward migration of the solution source, resulting in a downward shift of the whole vertical range of deposition during the time lapse between the East-West and Blue Vein periods of mineralization.

The cause of solution stoppage and subsequent renewal of activity poses an interesting but difficult problem, for the explanation of which the author has only a suggestion. It may have been caused by draining, through the East-West fissures, of the long accumulating reservoir of magmatic ore solution at a much more rapid rate than it was being supplied from the crystallizing magma. Under the hypothesis that the "ore solution" had become neutral or "acid" in final stages of accumulation, the effect of complete draining would be to restore the "magma residue" to a condition of feldspar stability, and this with decreasing pressure might cause crystallization of the granite minerals to continue to a point where the system again became a closed one. The processes of fractional crystallization, with its resulting magma residue, or "magmatic ore solution" accumulating within the *crystal mesh* beneath the newly formed solid cupola hood, would then continue at this deeper horizon just as it did at higher levels prior to tapping by the East-West fracturing, and without essential change in chemical composition.

The advent of Blue Vein fissures ended this period of ore solution inactivity. This fissuring resulted from changing stresses brought about by East-West fractures and the lessening of compressive strength of the intensely altered block of granite and porphyry, oblong in horizontal section and plan, reaching from the lower limits of East-West fissure penetration to the surface. Because of the increasing cross-sectional area of altered rock at successively lower horizons the Blue fissuring was centered over this roughly cone-shaped altered rock mass, and hence involved a much smaller area at and near the surface than was included in the earlier East-West area of fissuring. The Blue fracturing broke down through the newly formed barrier into the crystal mesh as far as fissurable and into the source of the newly generated ore solution.

The No. 16 vein, a northeast striking mineralized fault, closely followed the Blue veins in time, but all later faults, including the Rarus and Middle, either did not tap an ore solution source, or the source had receded to so great a depth that the deposited ores do not reach as high as deepest existing mine workings.

The Roles of Sulphur Silica and Water

Comparisons of certain characteristics of the Butte and Potrerillos types of copper deposits might be of interest. The Butte ores exhibit high sulphur, and much additional sulphur occurs as disseminated pyrite in the intense sericitic alteration zones encasing the ores, but orthoclase is absent as a vein mineral. The Potrerillos and Ajo deposits, omitting hydrothermal ore additions, show low pyrite, low sulphur, and very weak alteration of plagioclase feldspars, but large amounts of fresh orthoclase as a product of mineralizing activity. The primary copper minerals at Butte are chalcocite, bornite, enargite, digenite, covellite and chalcopyrite; at Ajo and Potrerillos they are chalcopyrite and bornite only. The absence of orthoclase mineralization is characteristic not only of Butte but of all high sulphur hydrothermal mineralizing activity in the porphyry coppers. The Butte zone of ore deposition is thought to be relatively far from the solution source, while the Potrerillos-Ajo type of orthoclase-copper mineral complex is believed to be a near source deposition. These facts are highly revealing as indicating, if not demonstrating, the tremendously important role played by sulphur in the processes through which the nature of an ore solution is determined at its magma source.

Sulphur, silica, and water are unquestionably the most effective agents of hydrothermal ore accumulation. Sulphur does not unite with silica to form rock minerals, but it readily forms compounds with iron and nonferrous metals common to ore deposits. As pointed out by H. D. B. Wilson in his interesting paper, Vogt's data on sulphide solubility indicate that 0.2 to 0.4 pct of sulphur can be dissolved in a basic magma, but sulphur is held only sparingly in magmas containing more than 55 pct silica. However, one of the most important questions relates to its disposal or distribution during solidification of the magma. It is highly probable that in the absence of water, sulphur combines with any copper, nickel, or excess iron present, to form an immiscible sulphide melt. During crystallization of the rock silicates the sulphide melt particles dispose themselves throughout the rock as disseminations, or they may sink and collect to form massive orebodies.

Under the foregoing premise it is apparent that a high-sulphur hydrothermal ore solution does not develop from a magma which crystallizes to form a basic rock, one of the reasons being that the basic

silicate minerals in such rocks are not carriers of free water and no other source is provided. It is known, however, that hydrothermal ore solutions are developed in granites, porphyries, and acid volcanic rocks, all having abundant free quartz as a rock mineral. Is it not reasonable then to look to a silica-rich magma as the most likely source of water at the critical moment of crystallization of the silica as quartz, for the creation of a hydrothermal ore solution, or the "magmatic ore solution" of Bowen and Ross?

Quartz was the last mineral to crystallize in the cooling phase of the quartz-monzonite at Butte. The question is: What was the physical and chemical make-up of the silica just prior to its crystallization as quartz? It could not have been pure liquid silica at the temperature of fluid granite, which many agree is several hundred degrees below the melting point of pure silica. It must have been something in the nature of a chemical compound, or physical combination of silica and water. Therefore, it is here suggested that at the moment of solidification as quartz the water was expelled to join the magma ore solution in its critical role as the carrier of the sulphur and other materials composing the magma residue.

At Sudbury a controversy of long standing had arisen between those supporting the theory that the massive sulphide bodies are accumulations of "magmatic" sulphides that have segregated immiscibly out of the basic norite magma and those advocating deposition of the ores from hydrothermal solutions derived from a magma source. Yates brings out the fact that two types of igneous rock are involved, the norite and a quartz-diorite. Is it not logical to conclude that both types of orebody could have developed: massive sulphides from the basic low-silica norite magma and hydrothermal ores along fissures from the high-silica quartz-diorite? The Mouat nickel-copper deposits in Stillwater County, Mont., associated with basic intrusives and granite, exhibit isolated copper-nickel sulphide masses in the basic rocks not related to fissuring, and a fissure vein containing lenses of sulphides similar in mineral composition to the massive sulphide bodies.

Summarizing, the author believes there is a series of events, fundamental in nature, that are essential to the build-up of copper deposits related to porphyries. These are: 1—a granitic batholith invasion of the earth's crust; 2—cooling of the batholith accompanied by processes of "differentiation and fractional crystallization" within the magma; 3—intrusion of a portion of the fluid magma upward into the earlier crystallized granite hood or into roof rocks, forming a localized sub-hood cupola; 4—crustal fissuring tapping the "magmatic residue", or "ore solution" developed within the cupola; and 5—formation of orebodies in association with the fissures.

The resulting type of deposit will depend primarily upon:

(a) timing of the "sub-hood" intrusion with respect to the stage of differentiation reached in the crystallizing magma below the granite hood;

(b) timing of the crustal fissuring which taps the "magma residue" accumulating in the sub-hood cupola; an early release producing mineralization of the orthoclase-copper type; a late release producing the high-sulphur hydrothermal types.

The "porphyry" and related copper deposits thus seem to be products of plutonic magmas containing excess silica. The more acidic, that is, the greater the excess of silica in a magma, the more water it will hold. Therefore, a granitic or quartz-monzonite magma should prove to be a favorable medium for supplying, during crystallization, water, sulphur and silica, the three substances essential to the accumulation of a magmatic ore solution.

At the beginning it was stated that a better understanding of the genesis of ore deposits is highly desirable as an aid to the maintenance of the nation's reserves of natural resources. Let it be suggested here, in connection with future laboratory research programs designed to add to existing knowledge of the genesis of sulphide deposits, that most serious attention and study be given, supplemented by experimental work, to the vital roles played by sulphur, silica, and water, in the development of ore solutions from magmas and in the distribution of ore minerals in zones of ore deposition.

Exploration of the Oaxaca Coal Fields In Southern Mexico

by Luis Toron and Salvador Cortes-Obregon

THE Oaxaca coal fields, shown in Fig. 1, are located in the Alta Mixteca region in the states of Oaxaca, Puebla, and Guerrero. Known outcrops are scattered over 3125 square miles. It is probable that further exploration will reveal a more extensive area of deposits.

The region is of great importance to Mexico, as at present the only sources of coal are the fields near the U. S. border. Steel mills are also located in the North, and finished products must travel 600 to 800 miles to the area of consumption in central Mexico. The Oaxaca coal, only 120 miles south of Mexico, D.F., lies close to several iron ore deposits. If this

L. TORON and S. CORTES-OBREGON, Member and Junior Member, respectively, AIME, are Mining Engineers, Estudios Geologico-Mineros, Banco de Mexico, S.A., Mexico, D.F.

Discussion on this paper, TP 3785F, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Feb. 18, 1953. Los Angeles Meeting, February 1953.

Fig. 1—On the above map of Mexico state boundaries are indicated by broken lines and railroads by solid lines. Locations of iron ore deposits are marked by heavy crosses, steel mills by triangles, and coal deposits by shaded areas.

coal can be used in steel production is will be possible for the Mexican steel industry to reach new dimensions, commensurate with the growing industry of the central region. Coal can be utilized in thermoelectric plants in this area, which lacks not only petroleum and natural gas but also electric power. It can be used as domestic fuel, replacing charcoal and firewood.

In March 1910 J. L. W. Birkinbine presented the results of his exploratory work in the coal region of Oaxaca.[1] His data were encouraging, but other workers[2-5] disagreed as to the value of the deposits. Studies were abandoned until 1949, when the present writers were assigned to evaluate the reserves and determine ways they might be utilized best.

Complete study of an area this size would cover a long period of time and would require the services of a great many specialists who are not available. For this reason it was decided, after initial reconnaissance, to concentrate on a zone with a large number of deposits close to existing communication facilities and to investigate the remaining area in the future. The selected portion of 120,000 acres is divided into two parts, the Tezoatlan-Consuelo area shown on the map and in cross-section, Figs. 2 and 3, and the Mixtepec area 30 miles farther south.

The study has been systematic, starting with detailed topographic and geologic mapping, followed by trenching in all the outcrops encountered. To determine continuity of the coal, samples were first taken in trenches, following procedures recommended by the U. S. Bureau of Mines. These samples gave results which vary considerably from samples of fresh coal taken from underground workings. At 15 strategic places along the outcrops drifts were driven 80 to 100 ft; samples were taken at points of farthest advance.

Table I shows analyses of 11 samples, selected for illustration from 250 samples taken on the outcrops.

Table I. Analyses of Samples Taken In Exploration of Outcrops[6]

Sample No.	Thickness Sampled, Meters	Dry Basis				
		Volatile Matter, Pct	Fixed Carbon, Pct	Ash, Pct	Calorific Value, Btu	Sulphur, Pct
66	1.00	19.50	26.26	54.25	4509	0.26
111	1.60	23.75	40.42	35.83		
113	0.70	19.97	45.35	34.68	7694	0.49
77	0.48	21.80	49.34	28.86		
3	2.20	17.82	48.44	33.74	8213	0.40
4	1.47	17.92	53.28	28.80	9120	0.43
213	1.48	13.78	47.57	38.65	7909	0.41
229	1.19	12.43	55.49	32.08	9311	0.47
14	1.42	7.63	43.25	49.11	7146	0.77
27	1.52	16.76	44.41	38.83	8523	0.64
42	3.70	18.45	46.40	35.15	8747	0.64

Ash content varies from 26 to 55 pct and heat value from 4500 to 9300 Btu. Table II gives results from two fresh coal samples, analyzed at the U. S. Bureau

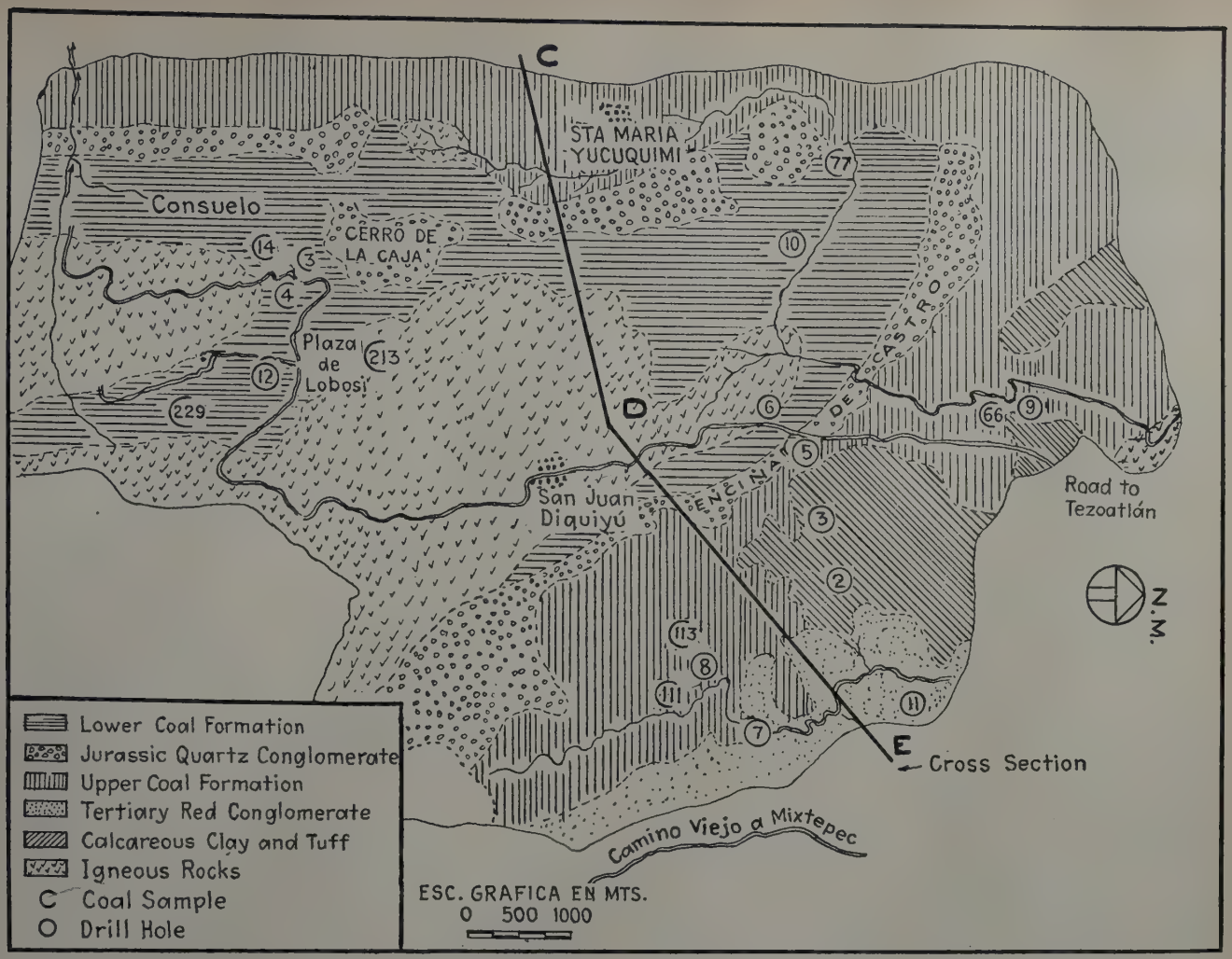


Fig. 2—Geologic map of the Tezoatlan-Consuelo area, Oaxaca. The Mixtepec area lies 30 miles farther south. The two regions make up the 120,000 acres selected for study.

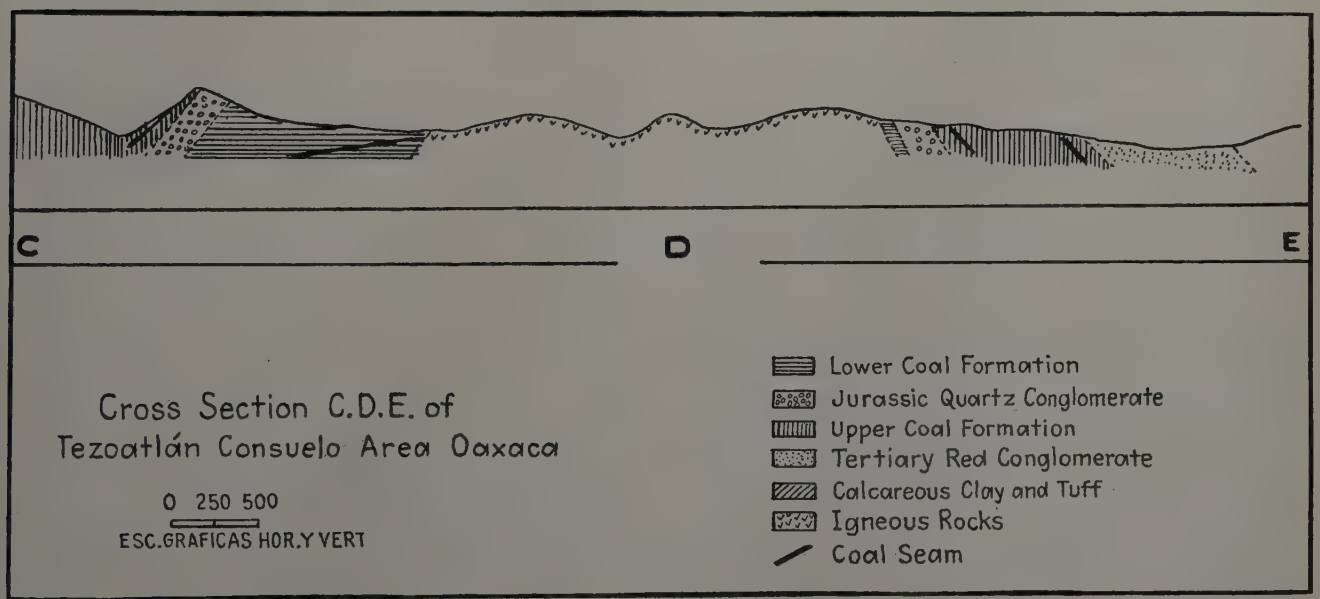


Fig. 3—In the Tezoatlan-Consuelo area, which received concentrated study, the stratigraphy is as follows: gneiss and schists; Triassic-Jurassic sediments, terrestrial, marine or lacustrine deposits, in which the coal is found; Cretaceous limestones; and finally Tertiary basalts and red conglomerates and andesite and rhyolite lavas and tuffs.

of Mines, which are typical of the two qualities of coal encountered in the 15 exploratory drifts. Ash content is 23 to 30 pct and the heat value, about 11,000 Btu, is much higher than that given in Table I.

The drilling program started in 1951 is continued today. Two 22 HD Joy rigs are being used, drilling 1100 ft with NX bit and 2500 ft with BX bit. Table III summarizes results of the drilling.

Simultaneously with the drilling program preliminary studies have been made on the quality, preparation, and utilization of the coal. At the Instituto Mexicano de Investigaciones Tecnológicas laboratory in Mexico, D.F., float-and-sink laboratory tests have been made, as well as carbonization tests on small samples. Other samples have been sent to the laboratory of Preparation Industrielle des Combustibles in France for similar analyses.

The area under study lies in the Sierra Madre del Sur, and its main surface characteristic is the great local relief, the elevations varying from 4500 to 7500 ft above sea level. The higher mountains have a sharp form and other secondary elevations a rounded shape due to stronger erosion. Mesas of great extent and almost flat summits remain as evidence of important lava flows.

The hydrographic system of the area is also complicated. During the rainy season numerous gorges discharge their currents into tributaries of the Balsas River, largest river on the west Mexican coast.

It is easy to understand that when this work was started, it was difficult to reach the areas under investigation. Long and tiresome rides on horseback were necessary to travel the region and to make the first reconnaissances. The Tezoatlan-Consuelo area is 15 miles south of Huajuapan, a village on the Pan-American highway, and the Mixtepec area 45 miles south of the village. From the Pan-American highway a primitive system of automobile roads has been developed, and it is now easy to reach all the workings by car and also to move the rigs to projected drill-hole locations. A station on the narrow-gage railroad is located on the Pan-American highway 100 miles north of Huajuapan. A standard-gage railroad crosses the highway 100 miles to the southeast of Huajuapan.

From the base upward,[8] stratigraphy of the zone is as follows: gneiss and schists; Triassic-Jurassic sediments, terrestrial, marine, or lacustrine deposits in which the coal is found; Cretaceous limestones; and finally Tertiary basalts and red conglomerates and andesite and rhyolite lavas and tuffs, see Fig. 3. In the Mixtepec zone there are also granitic and dioritic rocks.

The older Jurassic sediments are of the Lower Jurassic and can be identified by a rich flora of cycads that appears in several horizons, previously described,[7-9] which have provided a base for correlation. There are also well-preserved fossils of bivalves (Trigonia, Venus, Ostrea) and some genera of ammonites.

The Jurassic sediments, where the coal is found, have been sub-divided into three formations:

The lower coal formation, 1000 ft thick, is present only in the Tezoatlan-Consuelo area. It is composed of lutites, shales, and slates with layers of coal and carbonaceous shales, sandstones, and a dark brown conglomerate of limestone boulders held in a clayey cement.

The Jurassic quartz conglomerate, 150 to 300 ft thick, is found in both the Tezoatlan-Consuelo and Mixtepec areas. It is composed of some beds of quartzite intercalated with conglomerates of angular white quartz (also gneiss and schist fragments) cemented with a very hard siliceous material of red to yellow-brown color.

The upper coal formation, present in the Tezoatlan-Consuelo and Mixtepec areas, is over 1500 ft thick. It is composed of sandstones; mudstones and sandy shales with layers of coal and carbonaceous sandstones; slate; and thin layers of argillaceous limestone.

It should be pointed out that there are stratigraphic gaps in the area. The Upper Jurassic has not been identified, and limestone of Lower Cretaceous age has been found only in some marginal zones. It is not possible to state whether these stratigraphic gaps are due to interruptions in sedimentation or to intensive erosion of the post-Lower Jurassic sediments. On the other hand, a great part of the areas studied is covered by the Huajuapan formation, an interesting Tertiary formation[9] composed of a reddish coarse-grained conglomerate containing mainly limestone boulders, two thick basaltic lava flows, and a succession of sandstones and tuffs. This formation, partly eroded, covers the coal formations unconformably, as has been confirmed by the drill holes, and does not reveal the true extent of the coal formations or the possibility of a complete stratigraphic column at some place beneath the Huajuapan formation. Continuation of the drilling will solve these questions and others.

There are two workable seams in the lower coal formation with a thickness of 5 to 7 ft each, but in some sections only one of the seams has been found. The upper coal formation has two zones with workable coal. In each zone there are three seams, 5 to 7 ft thick, appearing in some places as two seams or as a single seam 10 ft thick or more, the others being absent or very thin.

The areas show considerable folding and some faults. It is difficult to establish a pattern that can be applied in detail to the strikes of the axes of the main synclines or anticlines. The tops of the anticlines have been eroded and the sedimentary formations can be seen only in places where the synclines have not been covered by younger rocks, mainly volcanics.

Table II. Analyses of Samples Taken In Small Mines Over 60 Feet From The Outcrop[6]

Item	D-87371 Lower Formation Zone of Tezoatlan		D-87370 Upper Formation Mixtepec Zone	
	As-received	Free from Moisture and Ash	As-received	Free from Moisture and Ash
Moisture	9		1.0	
Volatile matter	9.1	12.0	17.4	25.4
Fixed carbon	66.6	88.0	51.1	74.6
Ash	23.4		30.5	
Hydrogen	3.4	4.3	3.8	5.3
Carbon	68.3	90.2	59.0	86.0
Nitrogen	0.7	0.9	1.0	1.4
Oxygen	3.6	3.8	4.9	6.2
Sulphur as:	0.6	0.8	0.8	1.1
Sulphates	0.02	0.02	0.03	0.05
Pyritic	0.13	0.17	0.23	0.33
Organic	0.43	0.57	0.51	0.74
Btu	11560	15270	10430	15220
	Free swelling: non-coking		Free swelling index: 5	

The dip of the coals seams is variable, in general from 10° to 30°, with a tendency toward flatter dips at depth; in some places the seams dip from 30° to 50°. Consequently different types of mines can be predicted: almost flat mines and mines of moderate to strong inclination, with entrances by drift or by shaft. From the general information on small prospecting mines it has been found that the coal seam has one or two bands of bone which can be easily separated when the mine is worked. The roof is good and is expected not to present any problem.

Because the coal has a high ash content, as shown in Table II, some workers have called it black shale.[3, 4] The ash is finely and intimately distributed,

Table III. Data on Vertical Drill Holes

Hole No.		Coal Seams					Metamorphic Rock*	Bottom
1	From	55.33	65.08	70.00	240.28	324.96		
	to	57.26	67.00	70.86	242.50	325.96		369.72
	Thickness	1.83	1.92	0.86	2.22	1.00		
2	From	245.82	274.10	293.99	298.61	300.48		
	to	246.48	275.87	294.84	299.66	301.85		340.15
	Thickness	0.66	1.77	0.85	1.05	1.37		
3**	From							
	to		No coal					270.97
	Thickness							
5†	From							
	to		No coal				86.25	
	Thickness						116.44	116.44
6†	From							
	to		No coal				2.75	
	Thickness						295.14	295.14
7	From	110.66	130.80	173.12	179.51	194.51	260.85	
	to	112.03	131.77	174.03	182.06	196.29	265.79	
	Thickness	1.37	0.97	0.91	2.55	1.79		265.79
8	From	63.87	85.62	87.60			158.40	
	to	65.12	87.22	88.89			214.59	214.59
	Thickness	1.25	1.60	1.29				
9‡	From	99.02	169.51					
	to	99.78	171.41					179.31
	Thickness	0.76	1.90					
10	From	28.04	119.31				130.35	
	to	28.75	120.56				192.12	192.12
	Thickness	0.71	1.25					
11§	From							
	to		No coal					251.77
	Thickness							
12§§	From	23.57	26.94				64.41	
	to	24.54	28.46				87.56	87.56
	Thickness	1.07	1.52					

* An igneous metamorphic rock (basalt and andesite metamorphized).
** On a squeeze zone; no coal.
† To explore metamorphic zone; no coal.
‡ Continuance of drilling impossible because of bad ground.
§ Before coal seams were reached, continuance of drilling impossible because of bad ground.
§§ Horizontal strata.
All measurements are in meters. Thickness is measured along the drill and dip varies between 12° and 20°. Only seams thicker than 2 ft are tabulated.
No. 4 hole not finished at time of writing. Holes 5, 6, 10, and 12 are on the lower coal formation; the others are on the upper coal formation.

however, and the authors' own preliminary experiments carried out in the laboratory of Instituto Mexicano de Investigaciones Tecnológicas have proved that the coal can be washed in small sizes, —4 mesh. The tests made in France by Preparation Industrielle des Combustibles, a firm specializing in preparation of high-ash coal, have confirmed the results of the authors' experiments. The combined results of the testing programs have led to the following conclusions.

1—The coal from the lower coal formation is semi-anthracite, whereas coal from the upper formation is medium-volatile bituminous coal and shows considerable differences in the two areas studied. The coal from Mixtepec has good agglutinating properties and is suitable for the production of metallurgical coke. Coal from the Tezoatlan-Consuelo area, despite the fact that the seams have been correlated with the Mixtepec seams, does not have any caking property, possibly because of the closer presence of igneous rocks.

2—The fines can be washed by froth flotation. A product of 12 to 15 pct ash content has been obtained with 80 pct recovery.

3—The —½ in. coal has given a product of 12 to 15 pct ash content with 65 pct recovery.

4—No efficient cleaning can be expected with sizes larger than ½ in.

5—Because of the high quality of Mexican iron ores, the steel industry of the country can use coke with higher ash content than usual in other countries.

6—The high ash semi-anthracitic coal (23.4 pct ash and 11,560 Btu, as received) can be burned without washing.

7—Laboratory tests must be confirmed by large-scale tests.

At the present time, surface work and the drilling program under progress have confirmed a reserve of 100 million tons of coal. Mexico produces slightly more than a million tons of coal annually. Since the Oaxaca coal is of great importance to Mexico, it is necessary to continue the exploratory work to increase the reserves, and to investigate thoroughly the mining possibilities and the preparation and utilization of the coal.

References

[1] J. L. W. Birkinbine: Exploration of Certain Iron-Ore and Coal-Deposits in the State of Oaxaca, Mexico. *Trans.* AIME (1911) **41**, pp. 166-188.

[2] *Informe Final sobre los Trabajos de Exploración Ejecutados por la Compañia Minera "Donajhi," S. A.* (about 1928). Private report.

[3] H. Kipper, Director y Asesor de Mineria, de la Gutehoffnungshütte Oberhausen Aktiengesellschaft en Overhausen Rhld: *Informe sobre los Yacimientos de Carbón, de Minerales de Hierro, etc.* (1936). Private report.

[4] E. Diaz-Lozano and S. Villamar: *Informe sobre los Yacimientos de Fierro y Carbón en Las Mixtecas Poblana y Oaxaqueña* (June 1936). Private report.

[5] José A. Garcia: *Informe sobre los Yacimientos de Carbón de Consuelo y Mixtepec, Oax.* (April 1938). Private report.

[6] S. Cortés-Obregón: *The Coal Used in the Mexican Iron and Steel Industry.* Expert Working Group on Iron and Steel Industry in Latin America, United Nations, 1952.

[7] G. R. Wieland: La Flora Liasica de la Mixteca Alta. Inst. Geol. de Mex. *Bol. No. 31* (1914).

[8] G. R. Wieland: The Liassic Flora of the Mixteca Alta. *American Journal of Science Arts* (1913) **4**, No. 36, pp. 251-281.

[9] M. Maldonado-Koerdell: Nota sobre plantas fosiles del Rético de Ayuquila (Huajuapan de León), Oaxaca. *Bol. Soc. Geol. Mexicana* (1948) **13**, pp. 61-68.

[10] G. P. Salas: Bosquejo Geológico de la Cuenca Sedimentaria de Oaxaca. *Bol. Asoc. Mexicana Geol. Pet.* (December 1949) **1**, No. 2.

Block Caving Practice At The Jeffrey Mine

Large-scale caving operations in the asbestos orebody of the Jeffrey mine present problems in undercutting and slusher drift development. The solution of these problems and the development of aluminum forms for concreting slusher drifts are detailed in this three-part article.

BLOCK CAVING HAS BEEN DEVELOPED to a high degree of efficiency in the last two decades and more particularly since World War II. At the Jeffrey mine of Canadian Johns-Manville Co., in the Eastern Townships of Quebec, several innovations have increased production and decreased costs. Asbestos ore is produced at the rate of 10,000 tpd from an underground operation supplemented by a large open pit that has been mined since 1881.

The orebody consists of asbestos veins in a fractured and serpentinized peridotite. Hardness and structural strength of rock vary from soft shear zones at one extreme to a small amount of hard granitic inclusions at the other. In general the ore is weak and deteriorates on exposure to air, so that it is difficult to maintain underground openings for any period of time. Permanent workings are lined with concrete and temporary ones are driven minimum size and supported with steel sets as required.

The caving unit is a block 200 ft square, served by slusher drifts driven north and south halfway across the block from the haulage drifts. The average block is over 400 ft high and contains approximately 1.5 million tons. Caving is induced by mining out a horizontal slice above slusher drift points and across the bottom of the block. The units are prepared for undercutting as a number of small independent units which are grouped for simultaneous blasting to minimize effect of local ground failure in weak rock.

Aluminum forms are used for concreting slusher drifts, haulage drifts, and shafts, and advantages of speed in handling and safety due to their light weight promise to result in still wider application.

Part I

Slusher Drift Development

by H. H. Waller

ORIGINALLY slusher drift development was conventional, advancing the drift full 10x13-ft size at 6 ft per round. This proved dangerous and costly because the weak fractured rock of the orebody cannot be left raw for any length of time without its reacting with air to cause spalling. With this method of drift development ground conditions were worsened by many blasts and the length of time it was necessary for the drift to remain open.

The problem was practically solved by driving 5x7-ft pilot drifts, then enlarging to 10x13-ft raw size by slashing with 50 to 60-ft diamond drill holes. Calculated explosive charges are used to avoid wall and roof shattering. Still more recently jacklegs and sectional steel replaced the diamond drills, resulting in the same work for less than half the cost, see Figs. 1 and 2.

Crosscuts at right angles to the main haulage drifts, which cross through the center of the orebody, divide the ore into 200-ft blocks. Eight slusher drifts on 50-ft centers draw from one block. The slusher floor is 11 ft above the rail, inclined at 1 pct grade, and the top is 18 ft below the undercut. The finished slusher drift is 102.5 or 120 ft long, depending on whether it is north or south of the crosscut. Drawpoints are staggered on each side at 17.5-ft centers. The completed drift is concreted and has an inclined ramp and a vent at the back.

H. H. WALLER is Superintendent of Underground Mining, Jeffrey Mine, Canadian Johns-Manville Co. Ltd.

Discussion on this paper, TP 3767A Part I, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Dec. 9, 1953. New York Meeting, February 1954.

When crosscuts and gangways have been excavated and concreted and slusher stations installed, slusher drift development is carried out in 10 clear-cut stages: 1—Pilot drift is driven full length, 5x7 ft. 2—Station is excavated at half-way point in pilot drift. 3—Pilot drift is enlarged to slusher drift size by drilling. 4—Lower part of slash is blasted and mucked for floor concrete. 5—Floor is poured. 6—Inside upper half of slusher drift is blasted, mucked, and concreted. 7—Outside upper half of slusher drift is blasted, mucked, and concreted. 8—Drawpoints are excavated. 9—Drawpoints are concreted. 10—Concrete ramp and sheave protection recess are constructed at back end of slusher drift.

A 5x7-ft pilot drift is driven full length on the center line of the proposed slusher drift using a jackleg and detachable bits on ⅞-in. hexagon steel. A 10-hp air-driven slusher and 36-in. scraper pull muck directly into cars in the main haulage crosscut, see Fig. 2. Operations are on a three-shift basis. One man mucks the previous round, drills, and blasts his round in a shift. Average advance is 5 ft per shift.

When the pilot drift is completed, 7 ft of drift in the center section is enlarged to full raw slusher drift size of 10x13 ft, for the set-up to drill long slash holes.

The pilot drift is slashed to full raw size from this station with jacklegs and tungsten carbide chisel bits on ⅞-in. sectional steel. Crews line the holes without trouble, working from controls established by the engineers, and the pattern of holes for the slash is arranged to give the least shattering effect

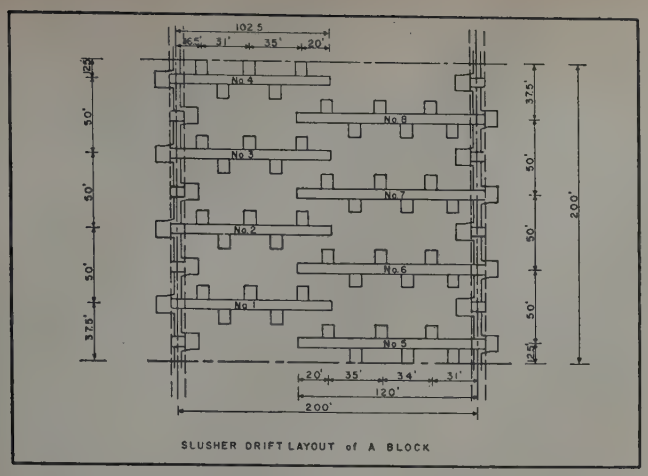


Fig. 1—Crosscuts divide orebody into 200-ft blocks, and eight slusher drifts on 50-ft centers draw from each block.

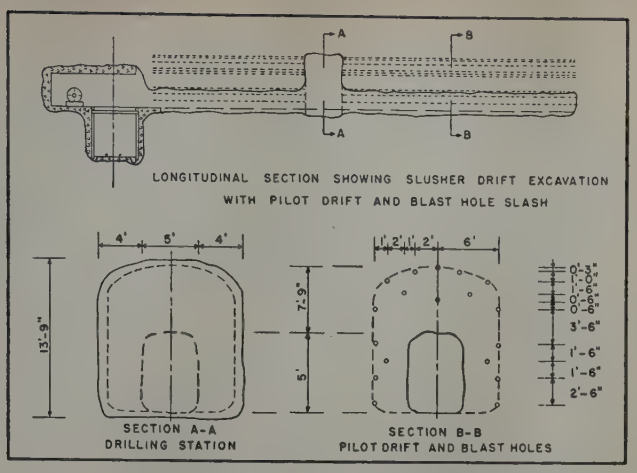

LONGITUDINAL SECTION SHOWING SLUSHER DRIFT EXCAVATION
WITH PILOT DRIFT AND BLAST HOLE SLASH
SECTION A-A
DRILLING STATION
SECTION B-B
PILOT DRIFT AND BLAST HOLES

Fig. 2—Sections show how long holes are used to slash 5x7-ft pilot drift to 10x13-ft raw slusher drift size.

Fig. 3—Concrete floor can be seen in view of slusher drift taken after blasting of upper half.

Fig. 4—In this photograph the aluminum forms are in place to concrete half the drift.

on the walls and roof from blasting. Two-man crews drill the 50 to 60-ft holes on line averaging 125 ft per man shift.

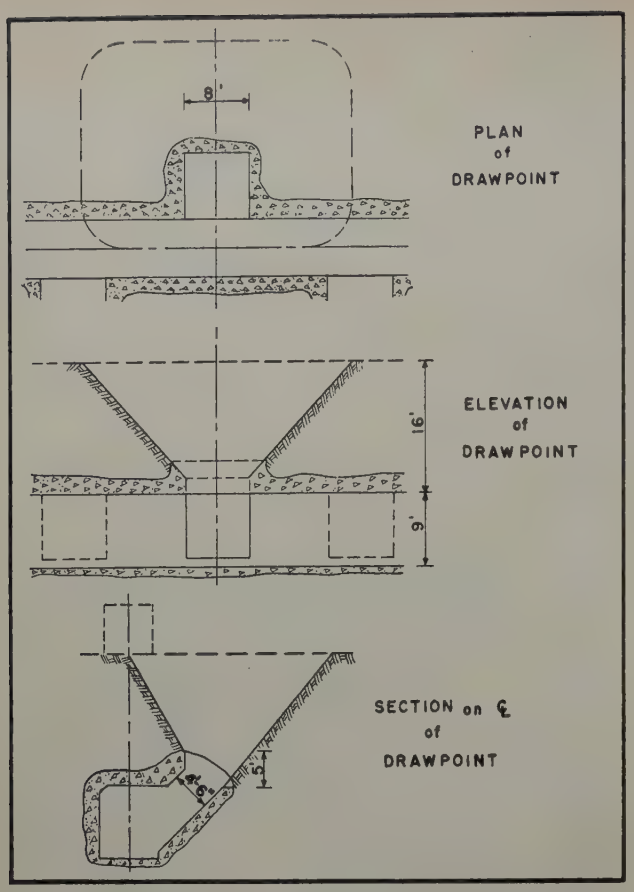
After drilling is finished, millisecond delay caps are used to blast the lower one-third full length. The slash is mucked directly into 10-ton tipple cars with a 42-in. scraper and a 15-hp air-driven slusher.

This excavation having been completed, a concrete floor 1 ft thick is poured the full length of the slusher drift. In the first 20 ft, 65-lb rail is placed in the concrete for wear protection. This concreting is done before the rest of the slash is blasted, because it is more economical to scrape the rest of the muck on a smooth floor, and the excavation provides the space required for blasting the remainder of the slash.

When the concrete floor is sufficiently cured, the upper part of the slash is blasted. Care must be taken with blasting, so millisecond delay detonators are used. Rotation prescribed by textbook is strictly followed to avoid any undue shattering of walls or roof. With these precautions, additional work can be continued without timbering, and this method of blasting also gives a well fragmented pile which is easily mucked into cars, see Fig. 3.

Before any more blasting is done, this opening or portion of the slusher drift is concreted, with recesses left at intervals for construction of drawpoints. Because of the quantity, concrete is poured on a mass production basis. Aluminum forms are used (*see Part III*) and concrete is placed behind them with a pneumatic Press-Weld placer. Concrete of at least 4000 psi (after 28 days and without reinforcing) is desired. A minimum of 18 in. of concrete is used. Reinforcing is not used because there has been experience of blasting vibration breaking the bond with the concrete and causing lines of weakness.

After the concrete has been poured 24 hr the second or front upper half of the slash can be blasted. The same procedure takes place here as in the first half. But in this operation one handling of forms for concreting does two jobs, namely stripping from the first half and placing again for the pour in the second half. It should be mentioned here that aluminum forms for concreting are particularly well adapted to handling. They are far superior to steel forms because they are light. Wood, of course, is not used as it would contaminate the fibre. At this point there is a concreted slusher drift minus



the drawpoints, but with recesses for them, see Fig. 7. Fig. 4 shows aluminum forms in place for concreting.

The drawpoints are among the last steps of developing a slusher drift. Because of bad ground condition it is necessary to develop the slusher drifts in sections and, for the same reason, to excavate and concrete the drawpoints separately. The excavation is made high enough to allow for an opening 8 ft x 4 ft 6 in. when concreted and at least 5 ft long. The drawpoint floor, at 45°, enters the slusher drift wall 1 ft above the drift floor, Fig. 5. Broken rock

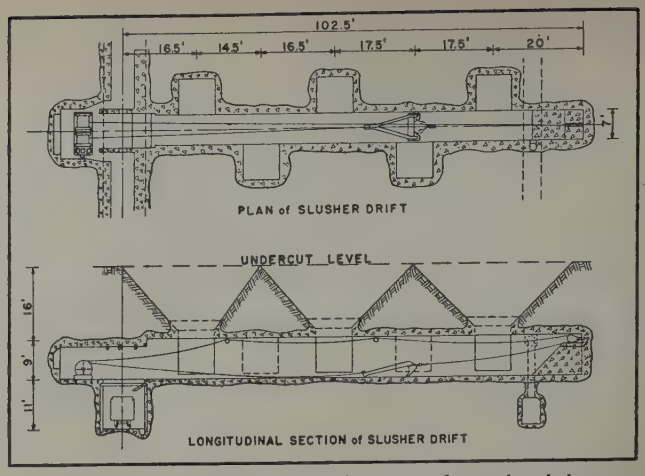


Fig. 5 (left)—Plan, elevation, and section of completed draw point. Fig. 6 (above)—Plan and section of completed slusher drift also shows ventilation arrangement located below drift.

is mucked with the regular production slushing unit, a 72-in. folding scraper and a 125-hp electric locomotive and 200 cu ft ore cars.

Concreting is done in much the same manner as in the slusher drift. The bolted aluminum forms are designed to allow the floor, walls, and brow to be poured in one operation. When using portable forms it is necessary to do a certain amount of bulkheading because it would be impossible to design a form to fit any rough wall, see Fig. 11, Part II.

The last part of the construction is a ramp to prevent the back end of the slusher drift from filling with muck which the scraper pulls back . The steel-faced concrete ramp at 45° slope extends to within 2 ft of the back end of the slusher drift and far enough ahead to allow a recess for sheave protection and to prevent the scraper from being pulled into the sheave, see Fig. 6.

The sheave saddle is a 4-bolt anchor set in the concrete at the end of the slusher drift approximately 6 ft above the floor. For ventilation purposes a 20-in. diam pipe is poured in concrete 11 ft from the end, so that it can be connected with the ventilation drift below. It is large enough to allow a man to travel through it in an emergency.

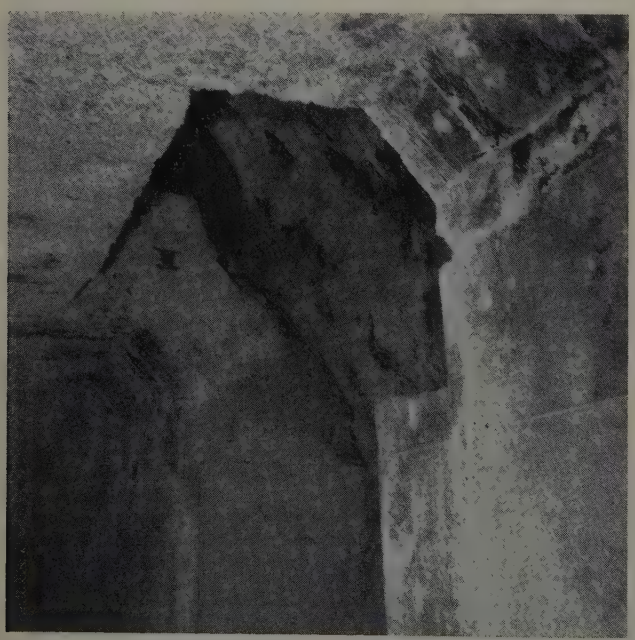


Fig. 7—View of concreted slusher drift shows recess left for drawpoint.

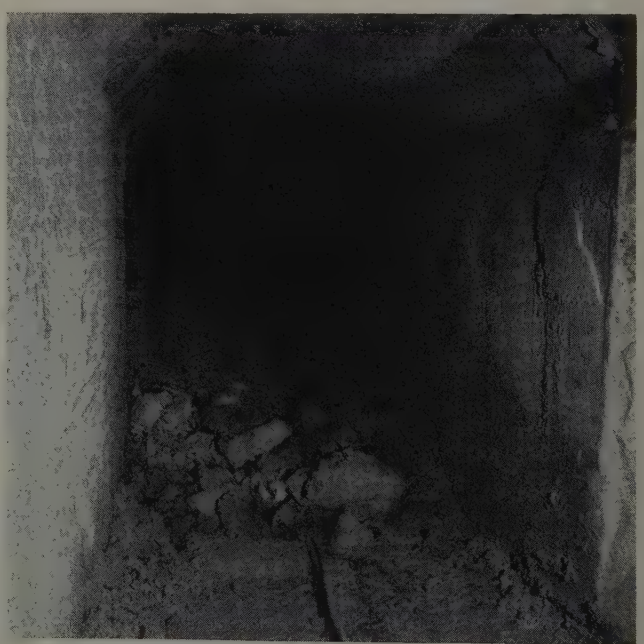


Fig. 8—Slusher drift, concreted as described, has required little maintenance.

The slusher drift is designed to stand a static head of at least 586 psi (500 ft of height) and to produce approximately 200,000 tons of comparatively soft rock. Although it does not often do so without some maintenance, it stands up well, and in some cases drifts have handled much more tonnage without any repair. Most failures come from point pressures which cause crushing. There is excessive wear on the floor at the delivery end and on the drawpoint brows, but this is easily repaired. At present, experiments are being carried out to ascertain to what extent heavy rails are necessary in the floor or, if they are desirable at all except for the first 20 ft at the delivery end. Steel rails were used at one time, but it was found that they were very little help in saving the walls. Fig. 8 is a view of the completed slusher drift in production.

There is no doubt that changes for the better will be made in this drift. At present, however, it is a very productive, low-maintenance slusher drift.

As concrete is used in such large amounts and so efficiently, it should be mentioned separately. Mixing is done on surface at a central automatic batching plant. Transportation is by shooting with a Press-Weld placer through a 6-in. pipe with compressed air for distances up to 2500 ft. Of this, 2000 ft is horizontal and about 500 ft is down a vertical shaft. Advantages of this method over the pumpcrete are many. Concrete can be placed at a much lower cost per yd, and a regular, more consistent mix is made. Congestion in the haulage crosscuts is also avoided because the concrete is carried in a 6-in. pipe either on the floor or back. Up to 80 yards have been poured in one shift.

Part II

Undercutting Technique

by D. L. Monroe

UNDERCUT development follows an advancing panel of slusher drift units 100x50 ft in area. This is further broken down into areas 35x25 ft over individual drawpoints, each of which is prepared for undercutting as a self-contained blast, and drawpoints for each slusher drift are grouped for simultaneous undercut blasting, Figs. 1 and 2.

The undercutting method now in use has served for the last six blocks with a minimum of modification. It is flexible enough to cope with all ground conditions encountered, except that extremely soft rock or extremely hard rock, and especially mixtures of the two, require special attention.

Undercutting consists of opening the bells over each drawpoint to the top of the undercut, wide enough to join each to its neighbor. This is done by opening a narrow vertical slot at right angles to the slusher drift and slashing parallel fans of holes to it to develop the combined drawpoint and undercut excavation. The success of the undercut depends on the slot. It extends to the margin of the undercut area and serves to outline the excavation as well as to provide the free faces and expansion for the final undercut blast, see Fig. 3.

D. L. MONROE is Chief Engineer (Design), Jeffrey Mine, Canadian Johns-Manville Co. Ltd.

Discussion on this paper, TP 3767A Part II, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Dec. 9, 1953. New York Meeting, February 1954.

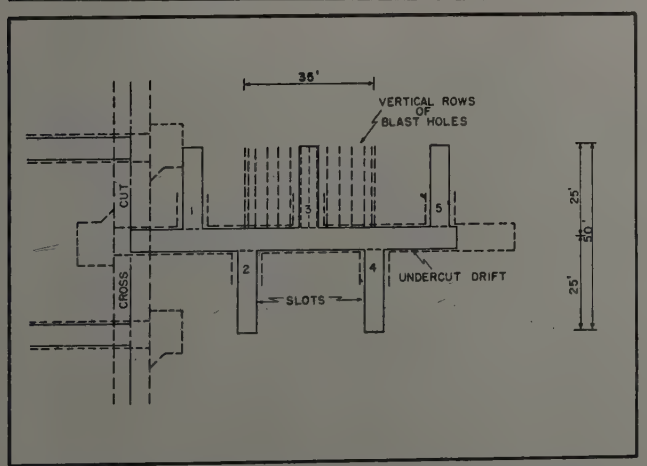
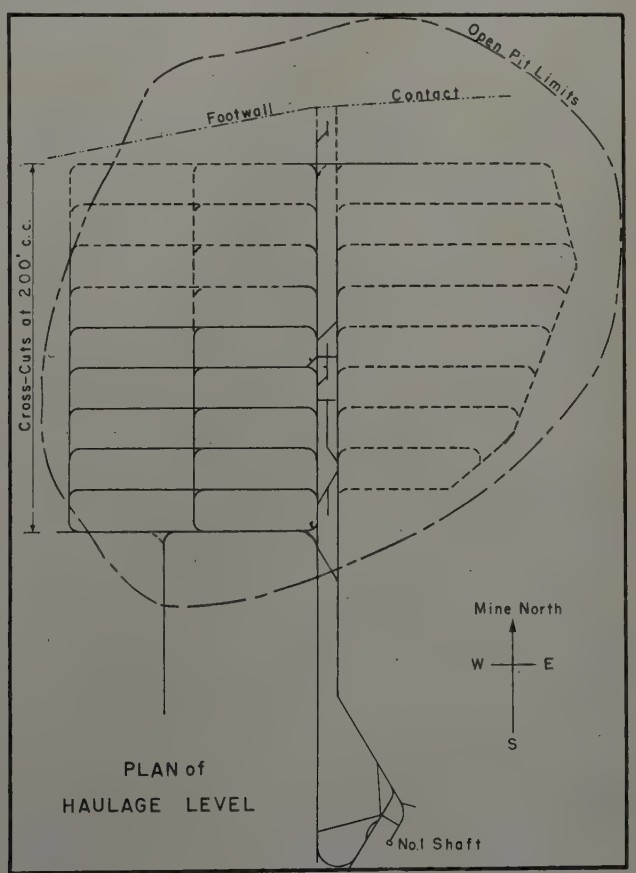


Fig. 1 (above)—Plan of undercut indicates blast holes for one drawpoint. Fig. 2 (right)—Plan of haulage level shows crosscuts that divide orebody into sections. Caving unit is 200 ft square.

Development

An undercut drift is driven directly above and parallel to each slusher drift. It is kept to a minimum cross-section and is opened no sooner than necessary to allow a continuous sequence of under-



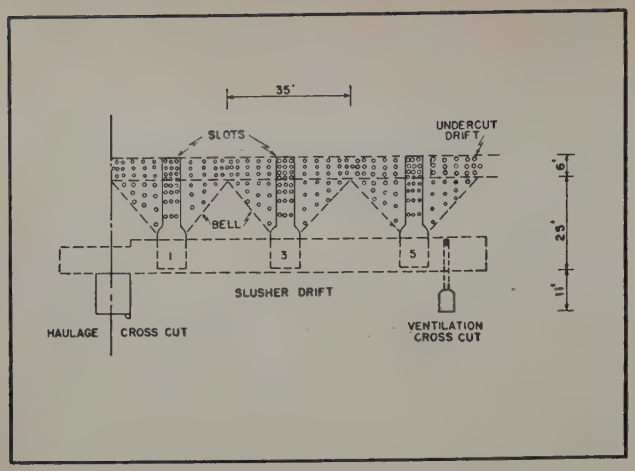


Fig. 3—Section shows placement of holes for drawpoint excavation. Drilling is with jacklegs.

cut preparation. It is often supported by steel sets and occasionally gunited as required. The undercut is drilled from this drift in a continuous pattern designed to progress from the bottom to the top of the slot and then out to the sides of the excavation. All drilling is completed before any blasting is started and, once started, blasting pauses only long enough to assure the complete breaking of the slot before proceeding with the rest of the holes.

The slot is opened by a footwall raise, a slot raise and the slot holes. The footwall raise is driven from the drawpoint to the undercut level at right angles to the slusher drift and establishes the footwall of the slot and of the future bell. This raise may be replaced by a pattern of footwall holes which, when blasted with the slot, are enough to clean up the footwall. This is all the work which is done from below, the rest being confined to the undercut drift above, as shown in Figs. 4 and 5.

The slot raise is drilled downward from the undercut drift to break through on the centerline of the drawpoint below. The slot raise and the footwall raise then form a vertical *V* at right angles to the undercut drift from the undercut down to the mouth of the drawpoint.

The slot is opened by blasting holes drilled from the undercut drift to break to the slot raise, forming a triangular slot extending from the drawpoint to the back of the undercut. Where footwall holes are used instead of the raise they are blasted at this time to clean the footwall.

The balance of the undercut drilling follows the pattern established by the slot drilling and consists of fans of holes at right angles to the undercut drift. Each fan contains holes drilled horizontally to the limit of the undercut area and downward to conform to each vertical section of the drawpoint bell. These rows are designed to be slashed to the slot, retreating from deepest near center to shallowest at the ridge between drawpoints, Fig. 5.

Each drawpoint is prepared as an independent unit to be blasted as part of a group to form a complete undercut across the area to be caved. Every drawpoint and every undercut prepared to date has continued to cave successfully.

Undercut drilling is now done with jacklegs using 7/8-in. hexagonal sectional steel and 1 3/8-in. tungsten carbide chisel bits. Holes are drilled up to 25 ft in length at averages of 125 ft per man shift with as much as 200 ft per shift having been reported. For the slot raise a *leyner,* sectional steel, and 3 1/4-in. and 2-in. steel bits are used, Figs. 6-11.

A 25-ft length of plastic hose is used for blowing holes, Fig. 7, and sectional plastic sticks are used for loading, Fig. 10. Polar Forcite Gelatin 40 pct and millisecond delay electric blasting caps are used.

A separate crew is maintained for undercutting and includes a blast hole foreman, an assistant foreman, who doubles as blast hole engineer, three shift foremen and about 30 miners. This crew operates as a unit and handles all phases.

Work follows prepared plans for drilling, loading, and blasting. The drilling plan serves as an indication of required departures from standard drilling patterns and as a check chart for holes drilled. The loading plan is a record of holes actually drilled, amended as required when the holes are blown, and on it are shown the powder and caps to be used. The blasting map is a completed loading plan on which all last-minute changes have been noted. The blasting circuits are worked out on this map ahead of time and are followed in the undercut drift when connecting up the blast. Records are kept of all phases of the undercutting operation up to and including actual date and time of final blast.

Advantages

Several advantages are claimed for this method. Safety to personnel is improved in that a minimum of drilling is done in and beneath the drawpoints and all other work is concentrated in the undercut drift. Control of drilling, loading, connecting, and

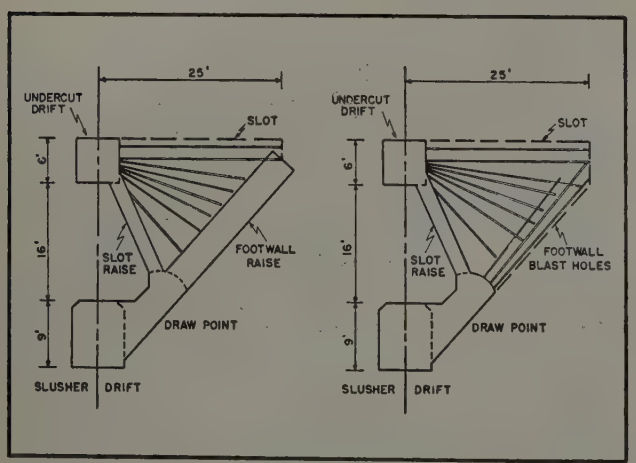


Fig. 4—Cross-section of slot development by footwall raise, left, and by blast holes, right.

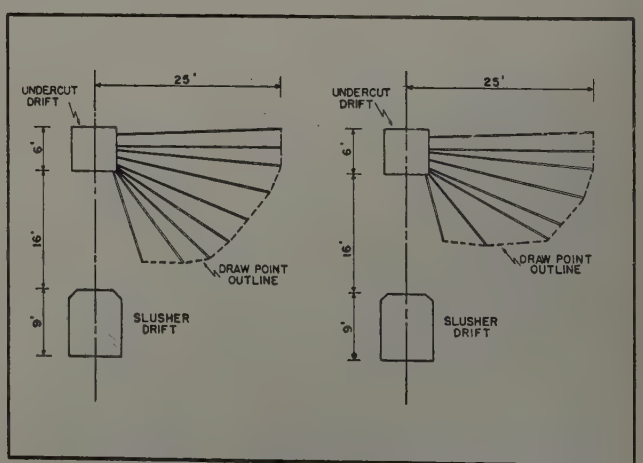


Fig. 5—Cross-section of first and second rows of the holes that slash to the already opened slot.

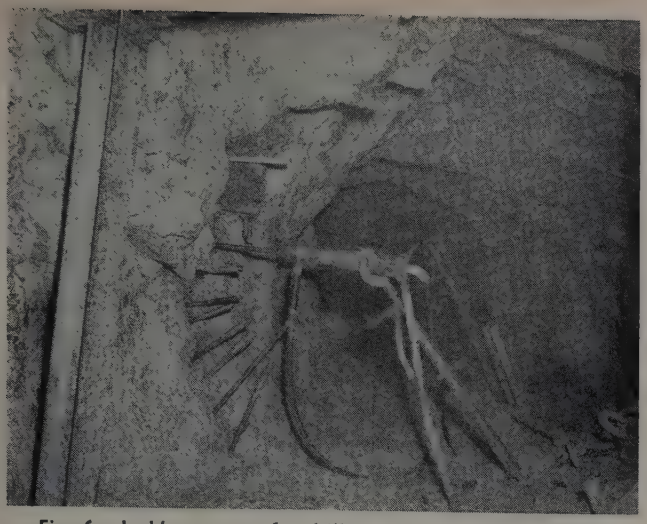


Fig. 6—Jackleg set up for drilling first row of holes from the undercut drift.

Fig. 7—This 25-ft length of plastic hose is used for blowing holes.

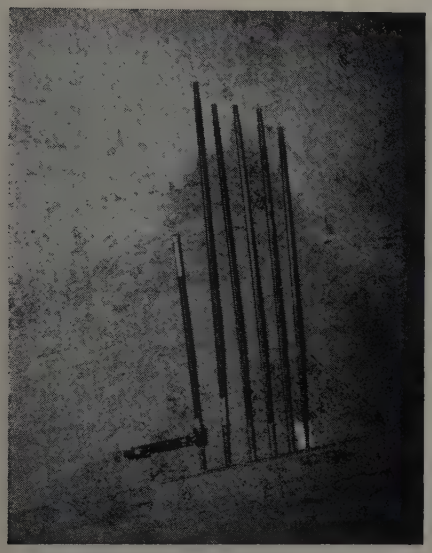


Fig. 8 (above, left)—Sectional steel is used for drilling up to 25 ft. Fig. 9 (above, center)—Leyner and sectional steel are used for the slot raise drilling. Fig. 10 (above, right)—Sectional plastic sticks speed loading. Fig. 11 (below)—View into mouth of concreted drawpoint shows footwall holes to be blasted with the undercut. Other holes are drilled from undercut drift above for maximum safety. Fig. 12 (right)—This undercut drift in bad ground is being supported with steel sets; gunite is also used occasionally. Drift is kept to minimum section and opened no sooner than necessary.

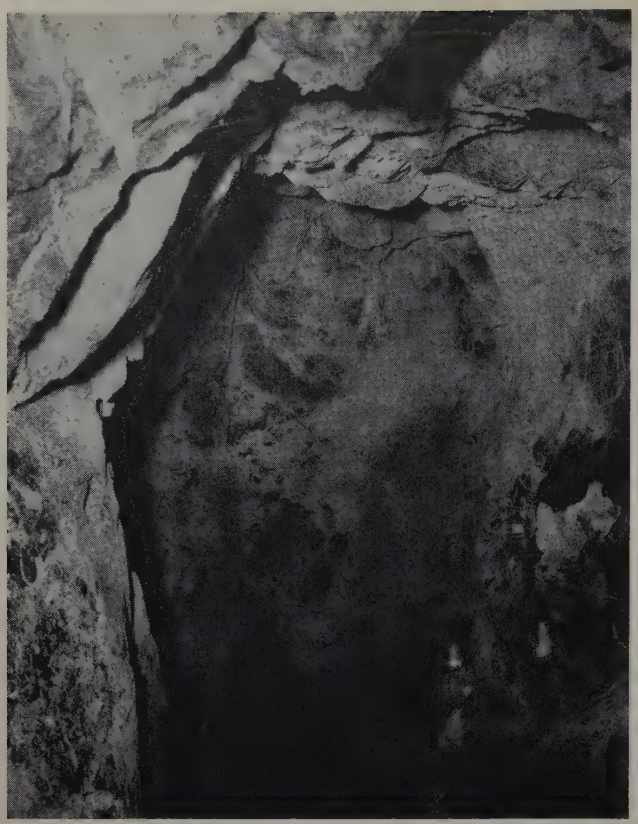


Fig. 14 (above)—Drawpoints in poor ground such as this receive individual attention.

Fig. 13 (left)—A view of completed slot in good ground.

blasting is facilitated in that work is laid out on plans and the whole undercut unit is prepared in one heading. Completion of an undercut, once started, is more certain in that a local ground failure, which is difficult to avoid entirely, need only affect the drawpoint in which it occurs. The hazard of ground failure is greatest at the junction of the slot and undercut drift. The slot is kept to a minimum width and is delayed for blasting as late as possible. Often the collars are left on the slot holes to bolster the undercut drift until the final blast. Instances occasionally arise where the slot cannot be maintained until blasting time and many holes are lost. Ground conditions then are usually so poor that the balance of the drawpoint will cave or be cleaned up by what holes may be salvaged. Drawpoints in such bad ground are identified beforehand and given individual attention, Figs. 12 to 14.

The undercutting method in use is well suited to average ground conditions and is flexible enough to allow occurrences of poor ground to be handled without altering plans for the whole undercut. It gives the greatest assurance possible under such varying rock strengths of a complete undercut across the area to be caved, and this is regarded as a prerequisite to successful operation.

Part III

Aluminum Forms for Concreting Mine Headings

by D. P. R. Smyth

ALL mine haulage and development openings at the Jeffrey mine are big enough for large-scale equipment. The ground is very weak. Permanent openings require concrete for support and aluminum forms are used for concreting slusher drifts, haulage drifts, and shafts. These forms improve the efficiency of concrete placement in all headings where they are used.

Roughly 65 pct of all mine concrete is poured in slusher drifts and draw points. Slusher drifts are located 11 ft above haulage drift rail base. Forms for slusher drift concreting must be moved by hand. It was early recognized that some kind of portable reusable forms should be used and that they should be made of the lightest possible material if labor costs were not to be excessive. Plywood forms were tried but after their second use some splintering of the forms was observed. Since in asbestos mining it is imperative that wood and other scrap be kept out of the ore, this splintering proved them unsatisfactory and attention turned to aluminum.

Aluminum Company of Canada Ltd. was consulted as to the suitability and availability of various aluminum alloys. It was decided that Alcan 65ST aluminum alloy would be used. This alloy belongs to the Al-Mg-Si series of alloys which are characterized by medium strength, good forming qualities, and good corrosion resistance. It welds satisfactorily, but that was not a deciding factor because the form design called for rivets and bolts only. Since then other designs have employed welding with very satisfactory results.

D. P. R. SMYTH is Supervising Mine Engineer, Jeffrey Mine, Canadian Johns-Manville Co. Ltd.

Discussion on this paper, TP 3767A, Part III, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Dec. 9, 1953. New York Meeting, February 1954.

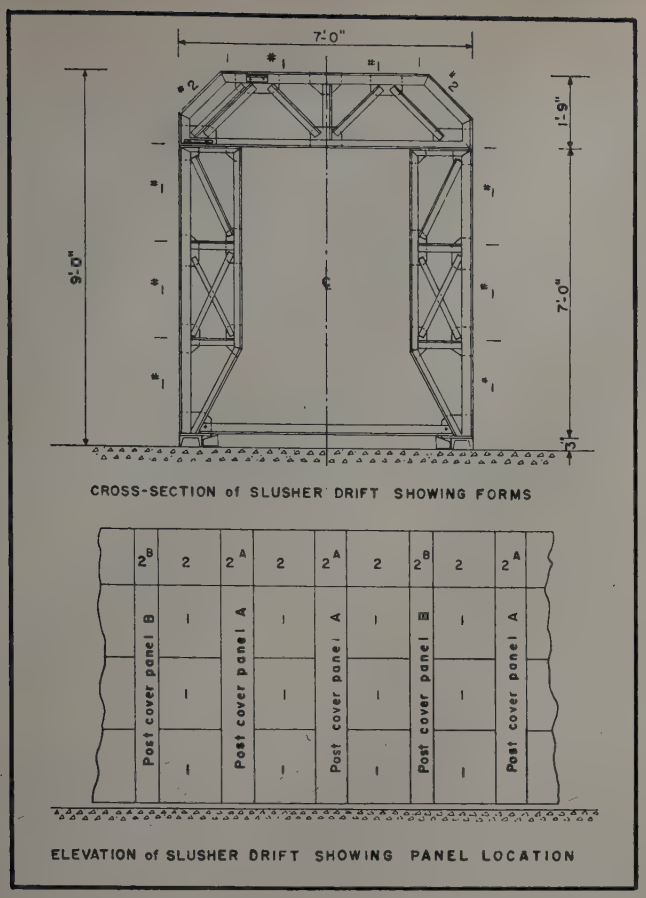


Fig. 1—Supporting truss for aluminum forms.

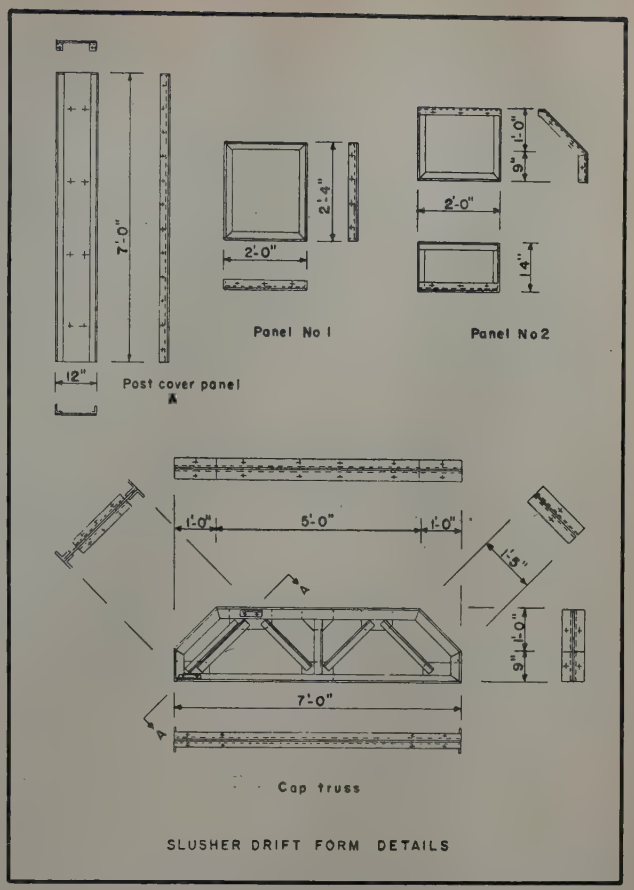


Fig. 2—Separate parts of slusher drift form.

Slusher Drift Forms

The cross-section of the slusher drift in Fig. 1 shows the supporting truss for the aluminum forms. Main members are made up of 3x3x⅜-in. angles riveted back-to-back to the gusset plates, while supporting members are 2x2x¼-in. angles similarly joined. The post truss is set on an inverted 6-in. channel and is wedged up slightly from the channel so that it can be more easily removed when the forms are being stripped. The elevation in Fig. 1 is a diagrammatic sketch showing where the panels are located in the slusher drift. Post cover panels are made in both 9 and 12-in. widths. No. 2B and No. 2A panels conform with the post cover panels.

The *cap truss*, detail, is in Fig. 2; see also Figs. 3, 4. When cap cover panels are bolted into place the nut should be in the concrete. Sometimes workmen forget this and put the nut on the inside of the forms. Stripping then becomes more difficult and forms may become twisted or bent. To obviate this abuse the cap truss is split as shown at section A-A on the drawing and can be dismantled in two parts.

The details of post cover panel A and of panels No. 1 and No. 2 are also shown; ¼-in. plate with 2x2x¼-in. angles is used throughout. The panels not shown are similar in construction. Except for post and cap cover panels, all bolting is done inside the forms.

Weights of the various panels are as follows:

Panel No. 1 and panel No. 2	25 lb ea.
Upright post truss without cover panel	75 lb
Cap truss	71 lb

Fig. 3 (above)—Close-up shows cap truss in place.

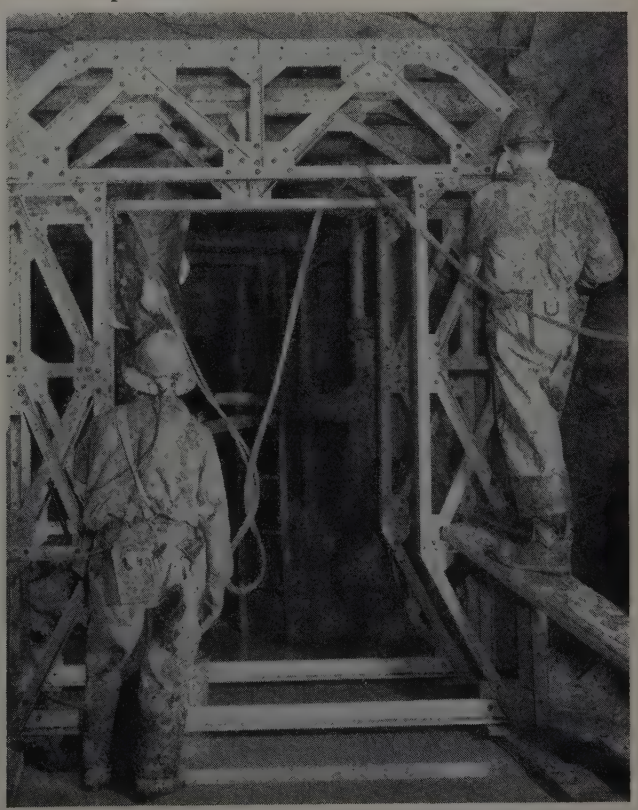


Fig. 4 (right)—Erection of slusher drift forms.

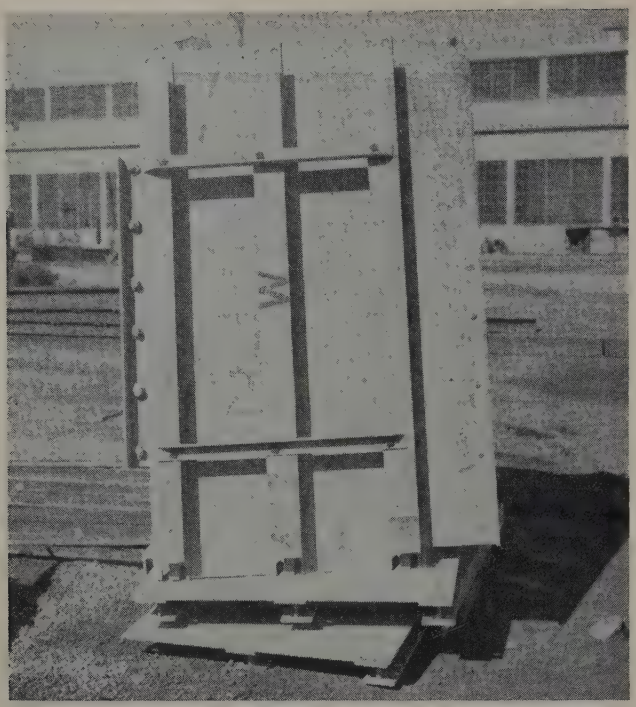


Fig. 5—These 4x6-ft aluminum shaft form panels weigh only 202 lb. They replaced steel panels weighing 611 lb and were used in sinking No. 2 main shaft.

Aluminum slusher drift forms have been in service for over 3½ years. As yet none has worn out. They are handled roughly and they have stood up to it. Some go to the shop to be straightened but there is very little breakage of member parts. A close examination of the oldest forms fails to find any pitting or corrosion. Steel forms used for concreting must be painted with lubricating oil before the concrete is poured or the forms will stick to the concrete very tenaciously. Aluminum forms are not painted and can be pulled off the concrete easily.

Experience with aluminum forms in slusher drifts led to their use in other headings. The No. 1 main shaft was sunk using steel forms for concreting. A steel panel 3 ft-10 in. x 6 ft-0 in. employing ¼-in. plate, 3x3x½-in angle, and 4x4x½-in. angle weighed 611 lb. Wider panels weighed proportionately more. All panels had to be handled by tugger hoist or other mechanical means. Aluminum panels, measuring 4x6 ft, now being used in No. 2 main shaft, weigh 202 lb. They are handled mainly by mechanical means but are much easier to place.

Fig. 5 is a close-up view on surface of one panel of the aluminum forms used in No. 2 main shaft. The 4x4-in. H-beams are riveted to ¼-in. aluminum plate and 2x2x¼-in. angles horizontal are added to facilitate handling the forms. Angles on each side of the forms vary to suit shaft timber or the next panel of forms. Alcan 65ST aluminum alloy is used throughout except that rivets are Alcan 55ST33.

In earlier mine development main haulage drifts were excavated in rectangular shape and timbered with steel sets placed at 6-ft intervals and then concreted into place. Forming for the concrete was done on the spot with steel plate and 4x4-in. steel H-beams. To obviate the use of steel sets and yet provide sufficient ground support a dolly-mounted arched-back steel form was designed. The bottom of the form is braced to the track and traffic can be resumed as soon as the form is placed, Figs. 6, 7.

Each 4-ft length of steel drift form weighs 1984 lb. A similar aluminum form weighs 612 lb. The final phase of levelling and lining up the forms is done by hand and the lesser weight of the aluminum makes it easier and safer to handle. The aluminum form also lends itself to use on reconstruction jobs where the dolly cannot go. The bottom section of the form is removed, the main section is divided in two by unbolting the top hinge and the largest resulting segment weighs about 215 lb which can be carried manually for short distances. The identical section of the steel form weighs about 700 lb and cannot be carried.

In an ordinary drift location, a crew working with the aluminum arched forms accomplishes from 40 to 50 pct more work than a crew does when erecting arched steel forms. Because aluminum forms for concreting have proved so adaptable and so economical of labor, the Jeffrey mine staff is planning to use them on all jobs where concreting can be standardized.

Fig. 6—Arched haulage drift form ready for concrete.

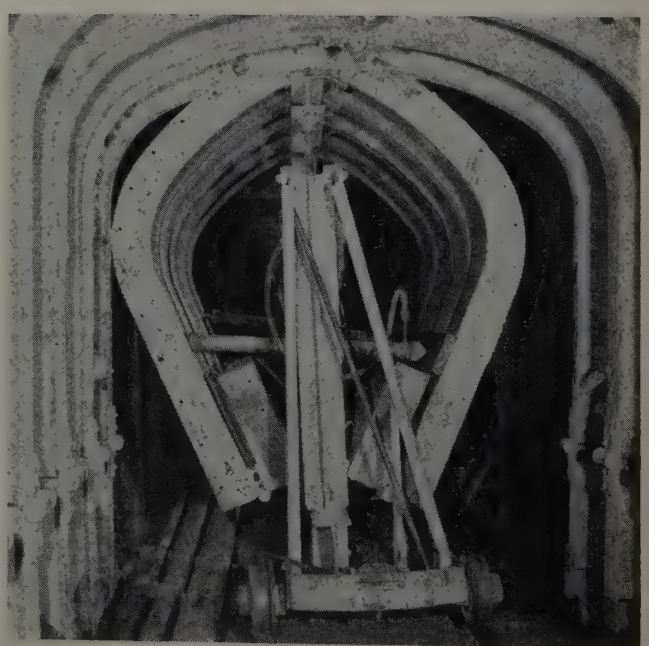


Fig. 7—Arched form being raised into place from dolly.

Flotation and the Gibbs Adsorption Equation

by P. L. de Bruyn, J. Th. G. Overbeek, and R. Schuhmann, Jr.

THE technique of concentrating valuable minerals from lean ores by flotation depends upon the creation of a finite contact angle at the three-phase contact, mineral-water-air. If the mineral is completely wetted by the water phase, contact angle zero, there is no tendency for air bubbles to attach themselves to the mineral. However, when the contact angle is finite, the surface free energy of the system, water-air bubble-mineral particle, can be diminished by contact between the bubble and the particle, and if not too heavy the mineral will be levitated in the froth.

With a few exceptions, all clean minerals are completely wetted by pure water. Thus the art of flotation consists in adding substances to the water to make a finite contact angle with the mineral to be floated, but to leave the other minerals with a zero contact angle. The contact angle concept and experimental measurements of contact angles have played important roles in flotation research for several decades.[1-3] Nevertheless, there remain unanswered some basic questions as to the scientific significance of the contact angle and the nature of the processes by which flotation reagents affect contact angles.

The contact angle is a complex quantity because the properties of three different phases, or rather of three different interfaces, control its magnitude. Considering the interfaces close to the region of ternary contact to be plane, the relation among the contact angle and the three binary interfacial tensions is easily derived. The condition for equilibrium among the three surface tensions, Fig. 1, or the requirement of minimum total surface free energy leads to Young's equation, Eq. 1:

$$\gamma_{SA} - \gamma_{SL} = \gamma_{LA} \cos \theta \qquad [1]$$

According to this equation, the contact angle has one well-defined value. Actually it is found in many experiments that the value of the contact angle depends on whether the air is replacing liquid over the solid (receding angle) or the liquid is replacing air (advancing angle). The receding angle is always the smaller of the two.[4]

Two explanations have been offered for this experimental fact. According to some investigators,[5-8] roughness of the surface causes apparent contact angles that are different for the receding and the advancing cases although the actual local contact angle may be completely determined by Eq. 1. The other explanation involves the hypothesis that the solid-air interface after the liquid has just receded is different from the same interface when no liquid has previously covered it.[1, 4] Adsorption of constituents of the air or liquid might play a role here.

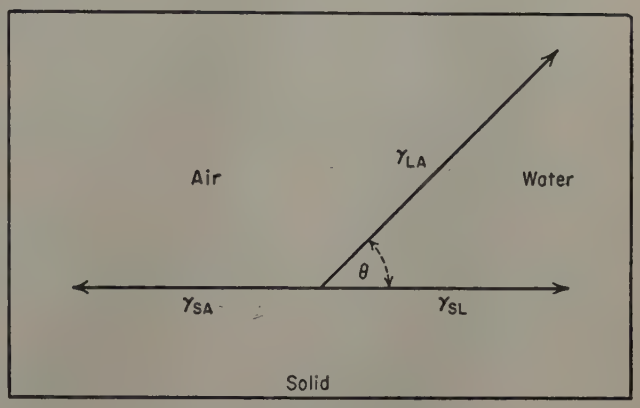


Fig. 1—The three-phase contact.

In this discussion the difference between advancing and receding contact angle will be neglected and plane surfaces where Eq. 1 describes the situation will be considered. But there is still a fundamental obstacle to the application of Young's equation. The surface tension of the liquid (γ_{LA}) can easily be determined, but the two surface tensions of the solid (γ_{SA} and γ_{SL}) cannot be measured directly. Eq. 1, however, is not without value. By contact angle measurements it is possible to establish how $\gamma_{SA} - \gamma_{SL}$ varies with the addition of solutes to the liquid phase. Also, Eq. 1 affords a convenient starting point for calculating net forces and energy changes involved in the process of bubble-particle attachment.[1, 9] If for the moment surface tension of the liquid (γ_{LA}) is considered a constant, an increase in $\gamma_{SA} - \gamma_{SL}$ will tend to decrease the contact angle. A decrease in $\gamma_{SA} - \gamma_{SL}$ corresponds to an increase of the contact angle. In cases where $\gamma_{SA} - \gamma_{SL} > \gamma_{LA}$ the contact angle is zero; it will only reach finite values when $\gamma_{SA} - \gamma_{SL}$ has been decreased below γ_{LA}. Thus on the basis of Young's equation and contact angle measurements alone, it can be learned how flotation reagents affect the difference $\gamma_{SA} - \gamma_{SL}$, but no conclusions can be drawn as to the effects of reagents on the individual surface tensions γ_{SA} and γ_{SL}, not even as to signs or directions of the surface tension changes resulting from reagent additions.

A quantitative relationship between the surface tension or interfacial tension and the adsorption occurring at a surface or an interface is given by the Gibbs equation, which for constant temperature and pressure reads

$$d\gamma = -\sum_i \Gamma_i \, d\mu_i \qquad [2]$$

where $d\gamma$ is the infinitesimal change in surface tension accompanying a change in chemical potential

P. L. de BRUYN, Associate Member AIME, is Assistant Professor of Mineral Engineering, Massachusetts Institute of Technology; J. TH. G. OVERBEEK, Visiting Professor at Massachusetts Institute of Technology (1952-1953), is Professor of Physical Chemistry, University of Utrecht, Netherlands; and R. SCHUHMANN, JR., Member AIME, is Professor of Metallurgical Engineering, Purdue University, Lafayette, Ind.

Discussion on this paper, TP 3743B, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Oct. 2, 1953.

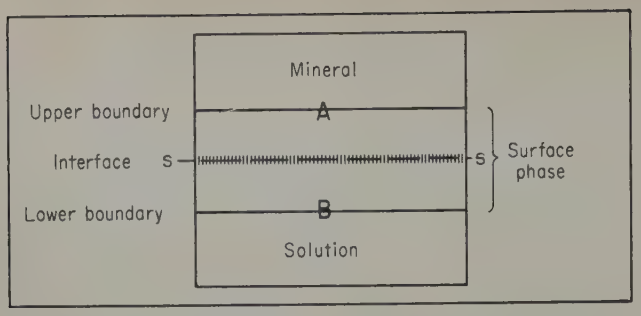


Fig. 2—The surface phase between mineral and solution. Planes A and B are on each side of the interface, sufficiently distant from the interface that the portions of the system not included between them can be regarded as completely homogeneous bulk phases. The mathematical dividing surface SS is usually chosen in such a way that the surface excess of the solvent is nil.

$d\mu_i$ of the component i of the system and Γ_i is the number of moles of component i adsorbed per cm^2 of surface.

In dilute solutions, the chemical potential of a solute is given by the expression

$$\mu_i = \text{const.} + RT \ln c_i \qquad [3]$$

where R is the gas constant, T the absolute temperature and c_i the concentration of component i in the bulk solution. The "const." depends on the solvent and on the units of concentration but not on the other solutes that are present in small amounts.

For the derivation of Eq. 2 the reader is referred to the literature.[4, 10, 11] If this equation is to be used intelligently, it is necessary to be explicit about the meaning of Γ and also to define correctly the components of the system to which the summation applies. Moreover, it should be emphasized that the Gibbs equation is applicable only when the surface is in equilibrium with the bulk phases.

Gibbs[10] pointed out in his fundamental treatise that the adsorption of any one substance cannot be defined in an absolute way. The adsorption of one chemical species can be expressed only in terms of that of another species. The amount adsorbed (Γ_i) may be defined as the excess amount of substance i present in the surface phase, that is, in the interface (or surface), plus the adjacent regions of the bulk phases, see Fig. 2. This excess is equal to the amount of substance i in the surface phase minus the amount of i which would have been present if the volume occupied by the surface phase were filled instead with the two homogeneous bulk phases separated by a mathematical surface. Defined in this way, Γ_i varies with the choice of position for the mathematical surface, since this choice determines the relative amounts of the two bulk phases which would theoretically have to be used to fill the volume of the surface phase. This uncertainty in the definition of Γ_i does *not* affect the value for the change in surface tension ($d\gamma$) in Eq. 2, because in the bulk phases the Gibbs-Duhem relation[12, 13]

$$\sum_i \Gamma_i \, d\mu_i = O \qquad [4]$$

is valid, where n_i is the number of moles of component i per unit volume. It is therefore immaterial where the mathematical dividing plane is placed or how much of the bulk phases is considered to belong to the surface phase. Often this plane is conveniently chosen so that one component, usually the solvent, is not adsorbed at all but belongs completely to the solution phase, see Fig. 2. This convention actually corresponds to the analytical definition of adsorption ordinarily used in experimental studies of adsorption from dilute solutions. Therefore, the summation in Eq. 2 must be extended to all components of the system including the mineral itself or to all components except the solvent if the assumption is made that the solvent is not adsorbed.

A further important point to consider is whether ions or neutral substances should be chosen as the components of the system. In principle this choice does not affect the final result, but since different ions as a rule are not adsorbed in equivalent amounts, it is often preferable to consider ions as the components. However, electroneutrality must be preserved both in the bulk of the solution and at the interface, and therefore the number of independent components is one less than the total number of ionic species present. Usually one or more relations exist among the chemical potentials of ions which further restrict the number of variables; for example, $\mu_{H^+} + \mu_{OH^-}$ is a constant and the solubility product of the mineral is a constant.

The relations can be handled very simply as follows: First write the Gibbs equation, Eq. 2, and consider the summation to be carried out for all ionic and neutral components except the solvent. Then introduce the principle of electroneutrality in the Γ's, use the relations among the chemical potentials to reduce the number of variables, and finally combine the chemical potentials of ions into those of salts, thus taking the electroneutrality of the solution into account.

Application to Flotation: Flotation systems usually contain such a large number of components that writing down the complete Gibbs equation for them, though not impossible or even difficult, would be tedious. Fortunately, in most cases what happens during simultaneous changes of all the chemical potentials is not of interest, but rather the effects produced by the addition of a single substance (collector, activator or depressant) to the system.

The Gibbs-Duhem relation, Eq. 4, shows that the chemical potential of *one* component cannot be altered without changing at least one of the others. In a dilute solution, however, the addition of a small amount of substance A to the system can be considered to change only the chemical potential of this component and that of the solvent. The chemical potentials of all the other components remain unchanged. Furthermore, the mineral is present as a separate phase and has, therefore, a constant chemical potential. When chemical reactions between the added substance and one or more of the other components occur, more complete forms of the Gibbs equation have to be considered.

The application of the Gibbs equation to a flotation system presupposes that equilibrium is attained or at least closely approached. Thus if the Gibbs equation is found consistent with experimental facts on actual systems, such agreement may constitute indirect evidence of attainment of equilibrium.

Influence of the Collector. A Simple Collection System: First to be considered is the addition of a collector that does not give rise to any chemical reactions or precipitations of insoluble phases. The solution already may contain any number of solutes which may or may not be adsorbed at the surface, but the concentrations and chemical potentials of these solutes will be kept constant.

If the collector is a neutral molecule, X, the Gibbs equation for addition of the collector would be

$$d\gamma = -\Gamma_X\, d\mu_X - \Gamma_{H_2O}\, d\mu_{H_2O} \qquad [5]$$

On the other hand, if the collector is an electrolyte, say C^+X^-, where X^- is the active collector ion and C^+ an inorganic cation like Na^+, H^+ or NH_4^+, the Gibbs equation will contain three terms,

$$d\gamma = -\Gamma_{X^-}\, d\mu_{X^-} - \Gamma_{C^+}\, d\mu_{C^+} - \Gamma_{H_2}\, d\mu_{H_2O} \qquad [6]$$

In Eqs. 5 and 6, the last term, $\Gamma_{H_2O}\, d\mu_{H_2O}$, is zero, if the convention is used that the adsorption of the solvent (Γ_{H_2O}) is zero. Usually, the second term in Eq. 6 can be neglected either because the adsorption of the cation (Γ_{C^+}) is small or because the concentration of the cation is already determined by the presence of an inorganic salt so that $d\mu_{C^+}$ is negligibly small.

It might be argued that if the collector ion is adsorbed by an ion exchange mechanism as demonstrated for chalcocite by Gaudin and Schuhmann[14] and for galena by Taylor and Knoll,[15] a term or terms for the exchanged anions ($SO_4^=$, OH^-, $CO_3^=$) should be added to Eq. 6. These terms, however, will be small, especially if the volume of the solution is large compared to the amount of mineral. The number of anions released to the solution by this ion exchange will hardly change the concentration of these anions in the solution; therefore, the relevant $d\mu$'s are very small and may be neglected.

In all cases of flotation, the collector is positively adsorbed to the mineral. This means that Γ_X or Γ_{X^-} is a positive quantity if Γ_{H_2O} is considered to be zero. Consequently, according to Eqs. 5 and 6, the addition of a collector *lowers the interfacial tension γ_{SL} between mineral and solution.*

According to Eq. 1 and Fig. 1 this decrease of γ_{SL} would lower the contact angle. Experiments, however, show an increase in the contact angle under these conditions. The experimental observation cannot be explained by an increase in the surface tension γ_{LA}, which, if affected, decreases slightly on addition of the collector. Neither can failure to reach equilibrium at the mineral-solution interface account for the increase in contact angle because this would only make the change in interfacial tension smaller and would not reverse its sign.

The conclusion is apparent, therefore, that in the collector system considered here the increase in contact angle has to be explained by a *decrease of the surface tension mineral-air* (γ_{SA}), a decrease which has to be larger than the decrease in γ_{SL}.

The decrease in the surface tension mineral-air is evidently brought about by adsorption of the collector to that surface. If the mineral-air interface is in equilibrium with the solution, the chemical potential of the collector should be the same at the mineral-air and the mineral-solution interface. A larger change in γ_{SA} than in γ_{SL} then demands a higher adsorption of the collector at the solid-air interface.

This analysis indicates that a collector should show not only a special affinity for the mineral, but in addition a tendency to prefer the mineral-air to the mineral-water interface. It should have, therefore, a strong hydrophobic group. As bases of an opinion on the qualities and possibilities of collectors, adsorption studies at the mineral-solution interface are certainly valuable and important, but for a good understanding of flotation more information on adsorption at the mineral-air interface seems indispensable.

Precipitate Formed by Collector. A More Complex System: Analogous conclusions are reached in the somewhat more complicated system where the collector ion forms a precipitate with one of the ions furnished by the mineral. Especially in xanthate flotation of sulphide minerals does this situation sometimes occur.[14] Taggart and Hassialis[16] have suggested that the solubility product of the compound between the collector ion and an ion from the mineral must be reached if collection is to occur. Without committing themselves to this point of view, the authors wish to show how the Gibbs equation must be treated in the case of precipitation.

Assume a system consisting of a mineral M^+A^-, a collector Na^+X^-, a salt Na^+B^- and water. The compound M^+X^- is assumed to be only slightly soluble and the amount of salt is large enough that addition of NaX does not change the chemical potential of the sodium ion. For the process of adding collector to this system the essential form of the Gibbs equation is

$$d\gamma = -\Gamma_{M^+}\, d\mu_{M^+} - \Gamma_{A^-}\, d\mu_{A^-} - \Gamma_{X^-}\, d\mu_{X^-} - \Gamma_{H_2O}\, d\mu_{H_2O} \qquad [7]$$

When both M^+A^- and M^+X^- are present as solid phases, the constancy of the solubility products can be expressed:

$$d\mu_{A^-} = -\, d\mu_{M^+} = d\mu_{X^-} \qquad [8]$$

With the assumption of zero adsorption of H_2O, Eq. 7 then becomes

$$d\gamma = +\,(\Gamma_{M^+} - \Gamma_{A^-} - \Gamma_{X^-})\, d\mu_{X^-} \qquad [9]$$

and with the condition of neutrality of the surface

$$d\gamma = (\Gamma_{B^-} - \Gamma_{Na^+})\, d\mu_{X^-} \qquad [10]$$

The value and even the sign of $d\gamma$ thus depend critically upon the detailed composition of the surface. If the adsorption of X^- is essentially an exchange with B^- and the adsorption of Na^+ is negligible, Γ_{B^-} will be positive as long as the exchange is not complete and Eq. 10 indicates that the interfacial tension *increases* with addition of collector. On the other hand, if the adsorbate is essentially Na^+X^- and the adsorption of B^- is small, Eq. 10 indicates that the surface tension *decreases* on collector addition.

In either case, however, the change in surface tension at the mineral-air interface is very important. If γ_{SL} goes down, γ_{SA} should go down more in order that a contact angle may develop. But, if γ_{SL} increases, γ_{SA} should increase more slowly. Assuming equilibrium to exist, this again means that Γ_{B^-} on the mineral-air interface should be smaller than Γ_{B^-} on the mineral-solution interface. In either case, an increase in contact angle results from collector addition only if the collector adsorption is greater at the mineral-air interface, just as in the simple collection system discussed earlier.

This example illustrates how important the determination of the adsorption may be, not only of the collector, but also of other components of the system.

Relation to Other Approaches: The usual explanation of collection is that the collector becomes adsorbed on the mineral surface with its hydrophobic end turned away from the mineral, thus leaving the surface hydrophobic or water-repellent. The same basic considerations are involved in this explanation

and in the approach based on the Gibbs equation. A collector should be easily adsorbed and should contain a hydrophobic part in its structure. The difference between the two approaches lies in the qualitative nature of the explanation based on the hydrophobicity of the surface as opposed to the quantitative character of the approach by the combined use of the Gibbs and Young equations.

Activation: An activator is a solute which promotes the flotation of a mineral by a collector which otherwise would have no effect on the mineral. A generally accepted explanation of activation is that the activating ion is adsorbed on the mineral and acts as a bonding agent between the mineral and the collector.

Several different forms of the Gibbs equation may be used to represent the effects of activators, and the choice will depend on what aspect of activation is being considered and on whether or not side reactions such as precipitation need to be considered.

First, if collection and activation occur without any precipitation reactions,[17] Eqs. 5 and 6, already discussed for simple collection systems, are still applicable in the presence of an activator. However, for minerals requiring activation, Γ_X or Γ_{X^-} and therefore $d\gamma$ resulting from an increase in collector concentration will be small or zero in the absence of the activator. A more informative approach to systems of this kind is obtained by writing the Gibbs equation for the process of increasing activator concentration, while keeping the concentrations and chemical potentials of the collector and of other solutes constant. To be specific, consider a system comprised of a mineral, a collector Na^+X^-, an activator Z^+Cl^-, and other solutes under conditions of no precipitation, complex-ion formation, or other side reactions.

The Gibbs equation for the process of adding activator then becomes

$$d\gamma = -\Gamma_{z^+}\, d\mu_{z^+} - \Gamma_{Cl^-}\, d\mu_{Cl^-} - \Gamma_{H_2O}\, d\mu_{H_2O} \qquad [11]$$

If chloride adsorption is negligible ($\Gamma_{Cl^-} = 0$) or if initial chloride concentration is large enough that the activator addition causes no appreciable change in chemical potential ($d\mu_{Cl^-} = 0$), and if the convention $\Gamma_{H_2O} = 0$ is used, the Gibbs equation for activator addition simplifies to

$$d\gamma = -\Gamma_{z^+}\, d\mu_{z^+} \qquad [12]$$

Thus, for this simple system in which it is postulated that no precipitation or other side reaction occurs, the addition of activator *lowers* the surface tensions of the solid. If the activator addition is effective in increasing the contact angle, clearly the adsorption of activator (Γ_{z^+}) must be greater at the mineral-air surface than at the mineral-solution surface. Since it would not be expected that activating ions by themselves would show any special tendency to accumulate at the mineral-air surface, it must be concluded that a greater adsorption of activator at the mineral-air interface is the result of the fact that the activator and collector are jointly adsorbed, each strongly affecting the adsorption of the other.

A common characteristic of activator-collector pairs is their tendency to react metathetically to form highly insoluble precipitates, and flotation has been observed under conditions where such reaction products are present.[18] For the system in which the collector Na^+X^- and the activator Z^+Cl^- react to form a precipitate ZX, the equilibrium of the solution with the precipitate requires that

$$d\mu_{z^+} = -d\mu_{X^-}. \qquad [13]$$

The Gibbs equation for addition of activator to this system then becomes

$$d\gamma = -\Gamma_{z^+}\, d\mu_{z^+} - \Gamma_{Cl^-}\, d\mu_{Cl^-} - \Gamma_{X^-}\, d\mu_{X^-} - \Gamma_{H_2O}\, d\mu_{H_2O} \qquad [14]$$

Omitting the terms for Cl^- and H_2O for reasons given earlier, and combining Eqs. 13 and 14

$$d\gamma = -[\,\Gamma_{z^+} - \Gamma_{X^-}\,]\, d\mu_{z^+}. \qquad [15]$$

If the activating ion has valence n and forms with the collector a precipitate ZX_n, Eq. 15 would become

$$d\gamma = -\left[\,\Gamma_{z^{n+}} - \left(\frac{1}{n}\right)\Gamma_{X^-}\right] d\mu_{z^{n+}} \qquad [16]$$

Eqs. 15 and 16 show that the direction of change of the surface tension on the addition of activator depends on the number of equivalents of activator and collector adsorbed. If the number of equivalents of activator adsorbed is greater than the number of equivalents of collector adsorbed, then the addition of activator is effective in reducing surface tensions of the mineral. Conversely, if the equivalents of collector adsorbed exceed the adsorbed equivalents of activator, the surface tensions of the mineral will be increased.

According to previously held views of the nature of activation, an effective activator is bonded both to the mineral and to the collector so that the valence of the activating ion must be divided between the collector and the mineral. That is, the number of equivalents of activator adsorbed is expected to exceed the number of equivalents of collector adsorbed. For example, Gaudin and Rizo-Patrón[19] have suggested that quartz flotation with oleic acid and barium chloride involves the adsorption of equimolar quantities of Ba^{++} and Ol^-, or two equivalents of Ba^{++} per equivalent of Ol^-. For this system, the expression in brackets in Eq. 16 will be positive and the effect of adding barium is to reduce the surface tension at both the mineral-air and mineral-liquid interfaces. To account for the increase in contact angle obtained by adding Ba^{++}, it must then be concluded that the adsorption of Ba^{++} and Ol^- or of the combination $BaOl^+$ must be greater at the air-mineral interface.

As more experimental data become available, it may become desirable to consider still other forms of the Gibbs equation for activation processes. For example, another possibility not considered above is an activator-collection system in which the activator and the mineral react to form a precipitate phase.

Depression: A depressant prevents flotation in a system where flotation would otherwise occur. The depressant therefore makes the contact angle decrease to zero.

A simple explanation of depressant action is obtained on the assumption that the depressant is adsorbed at the mineral-water interface but does not show any tendency to go to the mineral-air interface. According to this picture the depressant will lower γ_{SL} without affecting γ_{SA} too much. Such a behavior might be expected from hydrophilic, high molecular weight substances like starch which are used in potash and iron ore flotation operations.

Some electrolytes may act as depressants because they furnish ions which compete with the collector

ions and replace the collector at the mineral surface by ion exchange adsorption. In the absence of side reactions, the Gibbs equation for the process of adding depressants of this kind is essentially the same as for adding collector (Eq. 6 with X^- a depressing ion instead of a collecting ion) and leads to the reasonable conclusion that the contact angle is reduced if the depressing ion is more strongly adsorbed at the water-mineral surface than at the air-mineral surface. This results in the extremely interesting hypothesis that *both collectors and depressants may lower the surface tensions of both the mineral-water and the mineral-air surfaces, so that the difference between collection and depression, or between flotation and non-flotation, depends primarily on whether the added reagent tends to adsorb more at the mineral-air surface or more at the mineral-water surface.* It therefore becomes of great interest that experiments be devised to compare adsorptions at the two mineral interfaces, for depressants as well as for collectors.

Depression by deactivation, e.g., cyanide depression of copper-activated sphalerite, can be considered in terms of Eq. 12, provided the depressing agent is not itself also adsorbed on the mineral. That is, the depressant reacts with the activator (complex-ion formation or precipitation) to reduce the chemical potential of the activator. To refer to Eq. 12, $d\mu_{Z^+}$ will be negative and the changes in γ_{SL}, γ_{SA}, and the contact angle will be of opposite sign from those for activation when $d\mu_{Z^+}$ is positive.

Lack of Equilibrium at the Solid-Air Interface: Even if the mineral-solution interface reaches equilibrium readily, equilibrium is not necessarily reached at the mineral-air interface. The available data do not give any indication whether equilibrium is reached or not, except in so far as the difference found between receding and advancing contact angles suggests lack of equilibrium.

Under non-equilibrium conditions the straightforward application of the Gibbs equation is no longer possible. If, however, it is possible to determine the composition of the solution with which the surface would be in equilibrium, the surface tension could be calculated by assuming that the adsorption was built up in a reversible manner. The following integral then gives the surface tension

$$\gamma_{SA} = \gamma^{\circ}_{SA} - \sum_i \int_{\mu_i = 0}^{\mu_i^{eq.}} \Gamma_i \, d\mu_i \qquad [17]$$

where γ°_{SA} is the surface tension of the surface in the absence of adsorbing solutes ($\mu_i = 0$) and the integral is supposed to follow only equilibrium conditions. The upper limit of integration, instead of containing the chemical potentials in the actual solution, contains the chemical potentials $\mu_i^{eq.}$ corresponding to the actual adsorption. This integration requires information on the equilibrium adsorption isotherm, that is, data showing how Γ_i varies with μ_i under equilibrium conditions.

If adsorption equilibrium is approached both at the water-mineral and at the air-mineral surfaces under actual flotation conditions, it must be concluded that the adsorbed substances on both interfaces possess a high degree of mobility. The authors' analyses in terms of the Gibbs equation and the Young equation indicate that flotation depends on the existence of differences in adsorption densities between the part of the mineral surface in contact with air and the part in contact with water. Thus, as air spreads over a mineral surface, displacing water, the quantity of adsorbed collector should increase. The recent work of Hassialis and Myers[20] has shown a degree of mobility of adsorbed collector that may account for such an increase.

Conclusion

The principal value of applying the Gibbs adsorption equations to flotation systems is that the results point directly to the need for experimental work in several hitherto neglected areas of the surface chemistry of flotation. Above all, the relations developed by combining the Gibbs equation with the Young equation show that more attention should be given to the mineral-air interface.

To develop further the application of the Gibbs equation to flotation systems, simultaneous measurements should be made of contact angles and adsorption isotherms on both mineral-air and mineral-solution interfaces. Since it is probable that equilibrium at the mineral-air interface will be difficult to achieve and to measure, the use of volatile collectors, e.g., mercaptans, may be advantageous. Another possible experimental approach is to use a liquid *mineral* to obtain a system in which the three surface tensions could be determined directly. The system water-mercury-gas, with addition of collectors and other flotation agents, suggests itself for these measurements.

Finally, the question of attainment of equilibrium at the mineral-air interface highlights the desirability of further studies of mobility of adsorbed species, contact angle hysteresis, and related topics.

References

[1] E. D. Edser: British Association for the Advancement of Science, 4th Report on Colloid Chemistry (1922) p. 263.

[2] A. F. Taggart, T. C. Taylor, and C. R. Ince: *Trans.* AIME (1930) **87**, p. 285.

[3] I. W. Wark and A. B. Cox: *Trans.* AIME (1934) **112**, p. 189.

[4] N. K. Adam: *The Physics and Chemistry of Surfaces,* 3rd ed., pp. 107, 180. Oxford University Press, 1941.

[5] F. E. Bartell and J. W. Shepard: *Journal of Physical and Colloid Chemistry* (1953) **57**, p. 211.

[6] W. H. Coghill and C. O. Anderson: U. S. Bur. Mines *TP* 262 (1923) p. 47.

[7] R. Shuttleworth and G. L. Bailey: *Discussions of the Faraday Society* (1948) **3**, p. 16.

[8] R. N. Wenzel: *Industrial and Engineering Chemistry* (1936) **28**, p. 988.

[9] A. M. Gaudin: *Principles of Mineral Dressing,* p. 351. McGraw-Hill Book Co., New York, 1939.

[10] J. W. Gibbs: *The Collected Works of J. Willard Gibbs,* Vol. I, Thermodynamics, p. 219. Yale University Press, 1948.

[11] E. A. Guggenheim: *Trans.* Faraday Society (1940) p. 397.

[12] S. Glasstone: *Textbook of Physical Chemistry,* 2nd ed., p. 216. D. Van Nostrand Co., Inc., New York.

[13] G. N. Lewis and M. Randall: *Thermodynamics,* p. 207. McGraw-Hill Book Co., New York.

[14] A. M. Gaudin and R. Schuhmann, Jr.: *Journal of Physical Chemistry* (1936) **40**, p. 257.

[15] T. C. Taylor and A. F. Knoll: *Trans.* AIME (1934) **112**, p. 382.

[16] A. F. Taggart and M. D. Hassialis: *Trans.* AIME (1946) **169**, p. 259.

[17] A. M. Gaudin and C. S. Chang: *Trans.* AIME (1952) **193**, p. 193.

[18] R. Schuhmann, Jr., and B. Prakash: *Trans.* AIME (1950) **187**, p. 591.

[19] A. M. Gaudin and A. Rizo-Patrón: TP 1453, Mining Technology (1942).

[20] M. D. Hassialis and C. G. Myers: *Trans.* AIME (1951) **190**, p. 961.

Safety in the Mechanical Mining of Coal

by W. J. Schuster

Safety in coal mines depends largely upon adequate training of the foreman. Although management must provide modern and safe equipment and at all times keep mines in first class condition from a safety viewpoint, final results will be determined by the quality of supervision.

HANNA COAL CO., Division of Pittsburgh Consolidation Coal Co., operates three large underground mines in eastern Ohio. The section of Pittsburgh No. 8 coal seam in which these mines are located varies in thickness from 52 to 64 in. It is immediately overlain by a stratum of shaly material 12 to 15 in. thick locally known as draw slate, which is structurally very weak and which disintegrates rapidly upon exposure to atmosphere. Immediately above the draw slate as it is normally found is a band of extremely high ash material 6 to 12 in. thick known as roof coal or *rooster coal*, and above this is a stratum of conglomerate material varying from 4 to 10 ft in depth. Overlying the conglomerate is a relatively thick stratum of limestone, the first stable material above the Pittsburgh coal seam in eastern Ohio. With the method of full-seam mining that has been adopted, draw slate is shot down, loaded with the coal, and removed in the preparation plants. The roof coal then becomes the permanent roof.

The major problem in mining the No. 8 seam in eastern Ohio is control of the roof. Since the strata above the draw slate contains no material with a structure firm enough to provide self-support, the roof begins to sag in a relatively short time after the coal and draw slate have been removed. The problem thus becomes one of getting temporary safety posts under this roof as quickly as possible to prevent a break or separation from occurring either in the roof coal or in the conglomerate above it.

Haulage System

The Pittsburgh No. 8 seam in eastern Ohio is relatively level, with only minor local dips. Throughout the Hanna Coal Co. mines, entries are generally 12 ft wide. Rooms are driven on a 60° angle on 30-ft centers and are 22 ft wide. No attempt is made to extract the 8-ft pillars between.

The entire length of main line haulage is gunited in one mine, and a major portion in another. Two of the mines have single-track main haulage roads with passways. The third, a new mine, is double-tracked, and the roof is supported by steel crossbars, 60 lb or heavier, spaced on 4-ft centers and lagged. In recent years timbering on main line and secondary haulage roads has been accomplished by one of two methods: 1—crossbars are supported on a small section of post set in a hitch hole in the rib, or 2—or a hole is drilled in the rib about 12 in. below the roof, of sufficient depth to fasten securely a short length of 40-lb rail, the bottom of the rail facing the roof, on which a short post is set directly under the crossbar. At present the hitch-hole timbering method is favored.

At two of the mines the main line haulage locomotives are 26-ton, 8-wheel units. These locomotives are of the axleless type, each wheel being individually mounted on the frame. The motorman's compartment is encircled by 3-in. armor plate for the protection of the occupants.

At the third underground mine conventional 15-ton locomotives are being used. However, these locomotives have been completely rebuilt in the company's shops. Equipment has been streamlined and quarters have been provided for two people, who are protected by heavy steel plate in much the same way described above. This modernization program has been completed on all secondary haulage locomotives at the three mines, and the company is well on the way to similar equipment of the 6-ton section locomotives. The following additional features have been included in their modernization: 1—additional support for the motors to prevent their falling to the middle of the track and derailing the locomotives should a break occur in the suspension bar support; 2—installation of additional bracing to prevent brake rigging from becoming displaced and causing derailments; 3—enclosure of all electric wiring in conduit or raceway; 4—provision of an enclosed compartment for the storage of re-railers, jacks, and other equipment, so that they need not be carried on the outside of the motor; and 5—redesign of the end of the locomotive opposite the operator's compartment to prevent anyone's mounting from that direction. It is interesting to note that some

W. J. SCHUSTER is Safety Director, Hanna Coal Co., Division of Pittsburgh Consolidation Coal Co., St. Clairsville, Ohio.

Discussion on this paper, TP 3704F, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Aug. 26, 1953. Los Angeles Meeting, February 1954.

manufacturers turning out new locomotive equipment have adopted similar features.

Introduction of mechanical loading emphasized the need for high-speed haulage and made it obvious that safety measures must be taken to insure the safe movement of rolling stock in the mines. At the time mechanical loading was introduced the only control over the movement of trips was the telephone. Later hand-operated block signal systems were used. Both these methods, however, were seriously limited by human fallibility, and a few years ago the main line and secondary haulage locomotives and the supply and mechanics locomotives in the three mines were equipped with carrier-type radios known as *trolley phones*. The latest safeguards are the fully automatic block signal systems, which are laid out in these mines so that lights for illumination on haulage roads are off until a locomotive enters a particular block, at which time the trolley pole which actuates the block signal also turns on the lights along that signal block. When the locomotive leaves the block, the signal of course changes and the lights go off. The trolley phone and fully automatic block systems have considerably reduced the haulage accidents experienced under the telephone and hand-operated block signal systems.

Face Operations

All mining operations are performed with track-mounted mobile equipment. Room entry and room track use 40-lb rail on steel ties. Each room switch is equipped with a throw. In Hanna Co. mines permanent roof support is provided by steel crossbars, placed in position against the roof by mechanical timber-setting machines. These machines were designed by company engineers and built in the central machine shop under their supervision. Self-propelled, with provision for carrying a number of steel crossbars, they haul a truck containing wooden posts. The machines are equipped with mechanically operated lifting arms and an electric saw for cutting posts to the required length.

Compressed air is used for shooting. Air compressors are centrally located and air is piped to the working areas. Permissible powder is used only for such operations as shooting rolls in the bottom.

Post-mounted electric drills are used generally in company mines. Within the last few years these drills have been equipped with auxiliary foot or *dead man* switches, since there have been cases of serious injury to drillers whose clothing was caught in the rotating drill mechanism, the driller being unable to get back to the switch. The drill equipped with the dead man's switch operates only as long as foot pressure is applied on the switch, which has prevented several serious or fatal mine accidents.

Many far-seeing managements recognize that keeping their mines in first class condition from a safety standpoint also protects their investment and long-term efficiency of operation. They further recognize that if they expect their employees to get behind a safety program, they themselves, as managers and leaders, must be in moral position to earn the cooperation and safety-mindedness of the men who work in these mines. This is the crux of any successful safety program.

Coal mine accidents occur in coal mines. Prevention of accidents must therefore be carried out in coal mines. They cannot be prevented wholly in the office of a vice president, a safety director, or a superintendent. Accidents occur for one of the following reasons: 1—negligence of mine management in maintaining safety measures; 2—improper and/or inadequate supervision; 3—failure to train workers to do their work safely; 4—slackening of a worker's safety habits.

Importance of Supervision in Coal Mine Safety Programs

When management has done all it can to eliminate accident hazards caused by dangerous physical conditions in a mine, the next important consideration is the quality of its supervision. As a general rule, workmen are only as safety-minded as their foremen. Management must first inspire its foremen to good safety leadership, and this leadership requires the same personal characteristics needed to achieve consistently good production records. Fortunately the coal industry is emerging from the days when a foreman "bossed his men" to the day when the foremen gets his results by means of intelligent advice and supervision. If workmen have a wholesome respect for the man who is their leader, there is no reasonable goal in safety or production they cannot attain. As everyone knows, a good foreman is utterly fair with each of his men, has no favorites on the crew, willingly explains the reasons for his directions, does not ask his men to do any task he would not do himself, and displays sincere interest in the personal welfare of his men and their families. Unless coal management recognizes the importance of human relationships to the success of business enterprises, there can be little hope of any advance safety-wise.

The need for good foremanship manifests itself in the next essential for a safety program: training the men in safe work habits. Over a period of years men develop certain ways of performing their particular jobs which may not be good ones. When top management gets down to the grass roots of a safety program, it becomes the foreman's task to untrain the workman in these bad habits and retrain him in new ones. It is then that he will need the respect and friendship of the workmen, for unless he modifies his criticism with genuine praise he will be deeply resented. He must make his criticisms as candidly and objectively as possible. He must correct mistakes privately rather than embarrass the men before their fellow workmen. Even constructive criticism, however, will not be effective unless he is able to convince each man that it is for his own safety and the safety of his fellow workmen that he is attempting to help him.

Many people have wondered why a workman must constantly be reminded to perform his job in a safe manner, when his doing it in an unsafe manner might cause severe injury or death to himself. The answer is that human beings are fundamentally lazy and will take the path of least resistance. The foreman's task of establishing safe work habits does not end, therefore, with his initial training of the crew. Day after day he must supplement his instructions by constantly policing his section to make sure that his men are performing their work as safely as they can. This too requires that the highest degree of good relations exist between the foreman and the men he supervises.

Here, then, is where the safety program will either stand or fall. If right thinking and proper action are not achieved at this level, they will never be achieved at any other level. And if the foreman is expected to perform this colossal job, he must be prepared to

do it. There are still foremen who believe that the kind of management described above is merely coddling the men. Skill is required on the part of top management to convince them that sound principles of human relations will work in the long run, and there have been instances when foremen's reasoning will not accommodate principles of this kind. In such cases, the extremity of separating them from management has been resorted to. The majority, however, were convinced that their management was dealing with them as they were expected to deal with their workmen, and it was reasonable for them to believe that men working for them would wish to be treated in the same manner.

The foreman is aided by the safety engineer, who accompanies him periodically on inspection of his portion of the mine, pointing out to him where he has violated company safety policy, State mine law, and Federal law or code, watching closely the work habits of the men and bringing to his attention any sub-standard performances he observes. His report is made to the mine superintendent, and copies are sent to the general safety director and the vice president. The safety engineer leaves notes of his findings with the mine foreman and the section foreman concerned.

In these inspections the safety engineer tries to avoid giving the impression that he is checking up with the intention of finding fault. For some years, on the contrary, an award system has been set up for supervisors at Hanna Coal Co., based on their safety performance, i.e., the condition of the mine for which a particular supervisor is responsible, as well as the number of lost time accidents which have occurred to men under his supervision.

For the past several years individual awards have been given to each workman who has worked for a calendar year without a lost time accident. There are some mine managers who believe that a safety award system brings the natural result of a good accident record. At Hanna Coal Co. it is believed that there is no shortcut to safety but that such awards will be of assistance to the foreman, giving him a talking point in selling safety to his men.

One of the principal philosophies of good advertising is that the product to be sold should come before the public eye as frequently as possible. The same approach applies to the selling of safety. For this reason a safety slogan contest has been sponsored for several years as a supplement to on-the-job safety work. Company employees were invited to submit slogans, one of these to be chosen as the company slogan of the year. Prizes were offered for the best slogan submitted at each mine, and a grand prize for the best of all those submitted. These contests were very successful, not primarily because of the quality of the slogans submitted, but because they created a great deal of discussion. In many instances the whole family assisted in writing the winning slogan. Once this was chosen, it appeared as if by magic all over the mines, bulletin boards, and even automobiles. It is stenciled in Scotch light on locomotives, mine cars, and cutting and loading machines. Large Scotch-light reproductions are placed at strategic points in company parking lots, wash houses, and mantrip waiting stations. Everywhere the workmen turn their eyes they see "I O U SAFETY." It is also publicized in the company house organ.

A traveling trophy has been instituted and is awarded each year to the underground mine with the lowest accident frequency rate. This award is presented to the mine management and local union officials, including the safety committee, of the winning mine at the annual picnic held for all employees and their families. The coveted honor of taking home the trophy has created a competitive spirit among the underground mines in the interests of safety. Another highlight of the annual employees' picnic is the presentation to a U. M. W. employee of a new automobile. Eligibility for participation in the drawing for this car is that the employee must have been in the company employ for the entire year, with no lost time accidents. This occasion is further exploited by the foreman, who passes out to his men each quarter a ticket indicating that he has gone one quarter of a year toward eligibility for a chance on the car.

The company is endeavoring to start training of prospective workmen and foremen before it employs them by instituting mining courses in various high schools in the area. The courses include theory and practice of mine ventilation, mine drainage, roof structure and support, and other phases of mining technology. The company safety department keeps in close touch with these classes, frequently making its members available for lectures to the students, consultation with instructors, and tours through company mines for the purpose of acquainting the various high school mining classes with mining practices they have already learned in theory. Naturally, much of the emphasis in these classes falls on the subject of safety, particularly as it relates to such matters as mine gases, mine ventilation, and roof support. The classes indirectly help to promote safety among many present company employees. Time without number workmen have commented on questions put to them by their sons concerning mining and mine safety.

No discussion of safety would be complete without mention of the importance of the Miners Safety Committee in the company's program. When committees of this kind first became active, too often management resented interference with its prerogatives by anyone representing the union. At Hanna Coal Co., however, it is believed that if mine management is sincere in promoting safety it will welcome suggestions from any source whatsoever. It is true that at times a committeeman who is radical in his views will wrongly criticize what he feels is a violation of safety on the part of management. It has been found that many of these committeemen are sincere in their criticism but ignorant of what constitutes good mining practice. Their attitude often changes materially after a training course in some of the theory of coal mining.

To summarize, it must be emphasized again that safety in coal mines, where coal is loaded by machine or by hand, depends on the conditioning and training of the foreman. The training must be thorough and continuous. It must be planned on a level that will be easily understood by the foreman and attract his attention to the extent that he will be able to advise intelligently the men whom he supervises on safe work practices. So strongly is this true that even though management provides modern and safe equipment and excellent working conditions, employees must be adequately trained and supervised if accidents are to be reduced to the lowest possible minimum.

Evaluation of Sinter Testing

by E. H. Kinelski, H. A. Morrissey, and R. E. Powers

A group of 17 American blast-furnace sinters, an American open-hearth sinter, an American iron ore, and a Swedish sinter were used to evaluate testing methods adapted to appraise sinter properties. Statistical calculations were performed on the data to determine correlation coefficients for several sets of sinter properties. Properties of strength and dusting were related to total porosity, slag ratio, and total slag. Reducibility was related to the degree of oxidation of the sinters.

THIS report to the American iron and steel industry marks the completion of a 1949 survey of blast-furnace sinter practice sponsored by the Subcommittee on Agglomeration of Fines of the American Iron & Steel Institute. The use of sinter in blast furnaces, sinter properties, raw materials, and sinter plant operation have been reported recently.[1, 2]

After preliminary research and study,[3] test procedures were adapted to appraise the physical and chemical properties of sinter to determine what constitutes a good sinter. During the 1949 to 1950 plant survey each plant submitted a 400-lb *grab* sample to research personnel at Mellon Institute, Pittsburgh, Pa. A 400-lb sample was also submitted from Sweden. In addition, 2 tons of group 3 fines iron ore were obtained from a Pittsburgh steel plant. The following tests were performed on the iron ore sample and on the 19 sinter samples: chemical analysis; impact test for strength and dusting; reducibility test; surface area measurements, B.E.T. nitrogen adsorption method; S.K. porosity test; Davis tube magnetic analysis; X-ray diffraction analysis for magnetite and hematite; and microstructure.

Results of these evaluations are discussed in this paper and supply a critical look at testing procedures used to determine sinter quality.

Sinter Tests and Results

Each 400-lb grab sample of sinter was secured at a time when it was believed to represent normal production practice at each plant. It was not possible to use the same sampling procedures throughout the survey; consequently samples were taken from blast-furnace bins, cooling tables, and railroad cars. These were very useful for evaluation of test methods, since they were obtained from plants with widely divergent operations. With the exception of Swedish sinter and sinter sample N, which were produced on the Greenawalt type of pans, all survey sinters were produced on the Dwight-Lloyd type of sintering machines.

Sinters submitted for test were prepared in identical manner by crushing in a roll crusher (set at 1 in.), mixing, and quartering. To secure specific size fractions for tests, one quarter of the sample was crushed in a jaw crusher and hammer mill to obtain a −10 mesh size. The remainder was screened to obtain specific size fractions.

The group 3 fines iron ore was dried and screened and samples were taken from selected screen sizes to be used for various tests. Prior to testing, each ore sample except the −100 mesh fraction was washed with water to remove all fine material and was then dried. This iron ore, a hematitic ore from the Lake Superior region, was used as a base line for comparing results of tests on sinters. The iron ore did not lend itself to impact testing, since it was compacted rather than crushed in the test, and no impact tests are reported. However, the iron ore was subjected to all remaining physical tests to be described.

Chemical Analysis: Table I presents chemical analyses performed on the survey sinter samples. Included in this table are data obtained from determination of FeO and the slag relationships:

$$\frac{CaO + MgO}{SiO_2}$$ and total slag ($CaO + MgO + SiO_2 + Al_2O_3 + TiO_2$). The percentage of FeO was used as an indication of the percentage of magnetite in the sinter. It was believed that slag relationships could be correlated with sinter properties.

During initial determination of FeO great disagreement arose among various laboratories, both as to the results and the methods of determining values. Table I lists the values of FeO resulting from the U. S. Steel Corp. method of chemical analysis,[4] which reports the total FeO soluble in hydrochloric and hydrofluoric acids (metallic iron not removed) with dry ice used to produce the protective atmosphere during digestion. Use of dry ice was a modification required to obtain reproducible results. In this method, the iron silicates and metallic iron are believed to go into solution and are therefore reported as FeO. This is important, for in the study of the microstructure of sinters, glassy constituents suspected of containing FeO as well as crystallized phases of undetermined identity which may also contain FeO have been observed.

Strength Test by Impact: In evaluating sinter quality, one of the properties stressed most by blast-furnace operators is strength. This strength may be described as the resistance to breakage during handling of sinter between the sinter plant and the blast-furnace bins. It is also the strength necessary to withstand the burden in the blast-furnace. After

E. H. KINELSKI, Member AIME, is a Fellow of Mellon Institute, Pittsburgh, Pa. H. A. MORRISSEY, Junior Member AIME, was formerly a Junior Fellow, Mellon Institute. R. E. POWERS, Member AIME, formerly a Fellow, Mellon Institute, is now Chemical Engineer, Freyn Engineering Dept., Koppers Co., Inc., Pittsburgh.

Discussion on this paper, TP 3778B, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, Oct. 8, 1953. New York Meeting, February 1954.

Table I. Analyses of Plant Sinter Samples

Sinter	Chemical Analysis: Total Fe, Pct	Mn, Pct	P, Pct	SiO_2, Pct	Al_2O_3, Pct	CaO, Pct	MgO, Pct	S, Pct	C, Pct (By Combustion)	TiO_2, Pct	FeO Determination: Total FeO, Pct, Fe° Not Removed (HCl and HF)	Comparative Order: High, 1 Low, 10	Slag Relationships: $\frac{CaO + MgO}{SiO_2}$	Total Slag: $\frac{CaO + MgO + SiO_2}{Al_2O_3 + TiO_2}$
A	62.20	0.88	0.087	10.40		0.60	0.47	0.023	0.98		17.95	6	0.103	11.47
B	61.40	0.82	0.083	11.00		0.60	0.80	0.027	0.05		20.07	5	0.127	12.40
C	60.60	0.70	0.093	10.40		0.50	0.65	0.035	0.10		14.16	9	0.110	11.55
D	59.60	1.03	0.108	10.80		1.60	0.43	0.033	0.16		21.06	4	0.188	12.83
E	53.70	0.22	0.091	11.60		2.30	5.36	0.027	0.11		19.03	6	0.660	19.26
F	59.80	0.59	0.058	11.30	0.79	1.30	0.36	0.027	0.80		22.45	3	0.147	13.75
G	63.20	0.41	0.101	7.00		1.80	1.01	0.036	0.50		20.84	4	0.400	9.81
H	59.40	0.71	0.102	12.40	1.36	1.00	0.51	0.022	0.11		15.94	8	0.120	15.27
I	62.30	0.81	0.072	10.20		0.50	0.51	0.027	0.14		19.06	6	0.099	11.21
J	51.10	0.27	0.263	17.70		3.60	1.30	0.025	0.05		17.83	7	0.276	22.60
L_1	69.40	0.12	0.041	1.70		0.80	1.01	0.033	0.02		15.95	8	1.065	3.51
L_2	69.30	0.12	0.037	2.10		0.50	0.87	0.030	0.02		16.70	8	0.653	3.47
M	64.90	0.25	0.032	7.00	0.39	0.20	0.29	0.027	0.05	0.60	25.15	1	0.213	8.48
N	58.83	0.26	0.016	2.40	3.47	0.40	1.59	0.19	0.10	9.60	20.57	4	0.830	17.46
O	58.20	1.10	0.128	9.00		3.80	2.10	0.027	0.04	0.80	16.39	8	0.655	15.70
P	65.60	0.89	0.147	8.20		1.20	0.22	0.08	0.14		15.48	9	0.173	9.62
Q	65.40	0.62	0.201	7.60		1.10	0.43	0.05	0.10	0.30	17.88	7	0.201	9.43
Open hearth	66.60	0.40	0.250	4.40		0.40	0.36	0.05	0.10		24.42	1	0.173	5.16
Swedish	62.50	0.64	0.029	5.00		3.80	1.45	0.08	0.09		3.69	10 (very low)	1.050	10.25

extensive experimentation with the tumbler test, static crush test, and impact test, the impact test (adapted from one originating with the Jones & Laughlin Ore Research Laboratory[5, 6]) was found to be more suitable in the laboratory with regard to simplicity of operation, time required, and ease of representing strength values. The test was conducted on equipment illustrated in Fig. 1. After impact the sample (originally −1.050 + 0.263 in.) was screened and the percentages of +6 mesh (U. S. sieve size) and −20 mesh were taken as the two indices of strength (determined statistically as being suitable indices). While the indices were in most cases complementary functions and one screen size would be adequate for an evaluation, both sizes are given, Table II. The +6 mesh was arbitrarily called the strength index, since it measured the amount of strong particles that withstood the impact. Likewise the −20 mesh was arbitrarily called the dusting index, since it measured the amount of weak sinter pulverized during impact.

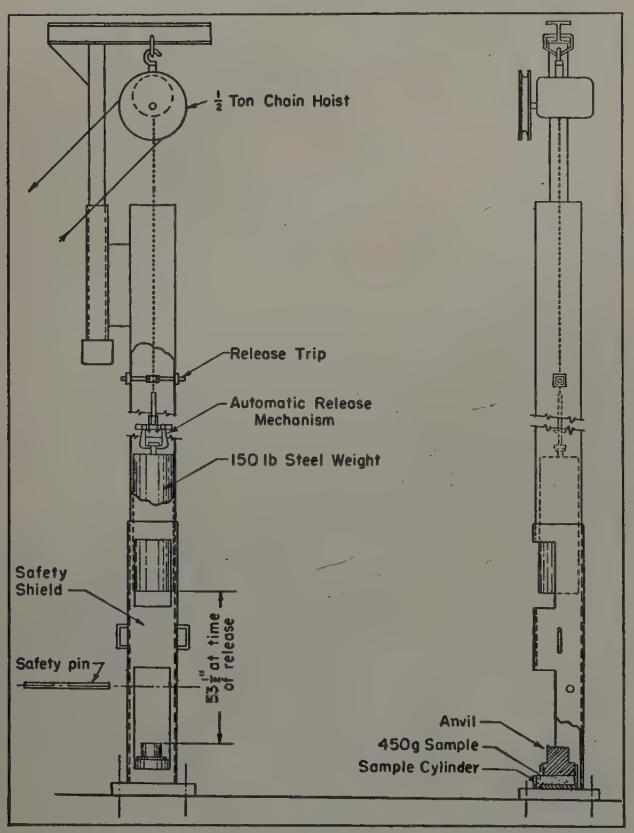


Fig. 1—Impact tester for sinter strength. After impact the sample (originally −1.050 + 0.263 in.) was screened and percentages of +6 mesh (U.S. sieve size) and −20 mesh were taken as the two indices of strength.

As evidenced by Table II, this strength index varied from 56.3 to 13.2 pct. Sinter samples A and E had the highest strength and sample L_1 the lowest. By comparison the sample of Swedish sinter had a value of 32.5 pct, approximately midway between the highest and lowest values. A comparative index from 1 for the highest value to 10 for the lowest value was established in an arithmetic sequence for the results in the tables.

Also in Table II, the lowest value of dusting was 15.2 pct, the highest value 34.8 pct. Furthermore, results show the least amount of dusting with samples A, B, and E, and the greatest amount with sample L_1 and the Swedish sample.

Table II. Impact Strength of Plant Sinter

Sinter	Impact Test Results: +6 Mesh, Pct	Comparative Order, Strength*	−20 Mesh, Pct	Comparative Order, Dusting*
A	53.9	1	15.8	10
B	47.1	3	16.0	10
C	46.3	3	19.6	8
D	47.7	3	18.7	9
E	56.3	1	15.2	10
F	42.4	4	20.6	8
G	41.8	4	20.1	8
H	42.5	4	19.1	8
I	51.3	2	17.4	9
J	47.0	3	20.1	8
L_1	13.2	10	34.8	1
L_2	23.8	9	29.8	3
M	35.0	6	28.2	4
N	41.7	4	24.2	6
O	44.7	3	17.8	9
P	48.8	2	18.1	9
Q	45.3	3	20.0	8
Open hearth	48.2	2	20.6	8
Swedish	32.5	6	33.2	1
Group 3 fines				

* High value, 1; low value, 10.

Reducibility Test: Perhaps the second most important property which blast-furnace operators

desire in sinter is ease of reduction. The test developed by Barrett and Wood[7, 8] was adapted and built as illustrated in Fig. 2. It was conducted at 800°C for 90 min, and loss in weight measurements were taken at intervals of 5 to 10 min. Temperature in the reaction chamber was then raised to 1000°C and reduction continued for 1 hr. A final loss in weight reading was then taken, and this value was used for calculating total oxygen available in the sinter to hydrogen reduction. Hydrogen flow rate was fixed at 0.124 cfm (STP) during the entire reduction.

In determining relative reducibility, loss in weight data was transferred into percentage reduction and plotted against time. The area under the reduction-time curve from 0 to 60 min was determined by means of a planimeter. This area was reported as a percentage of the similar area determined for group 3 fines iron ore, selected as a base line for the comparison of samples.

As Table III demonstrates, the sample of Swedish sinter had the highest relative reducibility, with a

Table III. Hydrogen Reducibility of Plant Sinters

Sinter	Reducibility		
	Oxygen Available, Pct	Relative Reducibility, Pct	Comparative Order* of Reducibility
A	23.80	88.4	3
B	24.10	81.5	4
C	24.07	92.0	2
D	22.86	82.5	4
E	20.67	79.0	5
F	23.38	79.8	5
G	24.02	67.4	7
H	23.55	84.2	4
I	23.18	76.4	5
J	19.03	88.9	3
L_1	26.07	87.7	3
L_2	26.33	84.8	4
M	23.37	71.5	6
N	22.32	51.1	10
O	22.44	81.4	4
P	24.30	88.8	3
Q	24.00	91.0	2
Open hearth	25.90	83.9	4
Swedish	25.10	98.1	1
Group 3 fines	28.90	100.0	1

* High value, 1; low value, 10.

value of 98.1 pct, as compared with group 3 fines ore. The best reducibility for all samples of American sinter was 92 pct as determined for sinter sample C. The order of reducibility extended from 98.1 pct down to 51.1 pct, the latter being for sample N.

Surface Area Measurements: In assessing the properties of blast-furnace sinters, mention is often made of surface area available for gaseous reduction. B.E.T. nitrogen adsorption measurements[9] for determination of surface area have been conducted, on −0.093 + 0.065 in. sieve size fractions, and results appear in Table IV. Of special interest is the tremendous difference in surface areas between sinter and group 3 fines iron ore, the latter being 89,000 sq cm per g, while highest surface area for sinter was on sample A with 3500 sq cm per g. Lowest area for sinter was 800 sq cm per g for sinter sample N.

The true or particle density of the material is also obtained with the B.E.T. equipment. As given in Table IV, the densest samples were found to be L_1 (5.09 g per cc) and L_2, while the lightest sample was sinter J (3.70 g per cc).

Porosity Test: An evaluation was made of the S.K. porosity test for determining porosity of sinter. Test equipment and procedures were adapted from those used by the Oliver Iron Mining Div. laboratories of

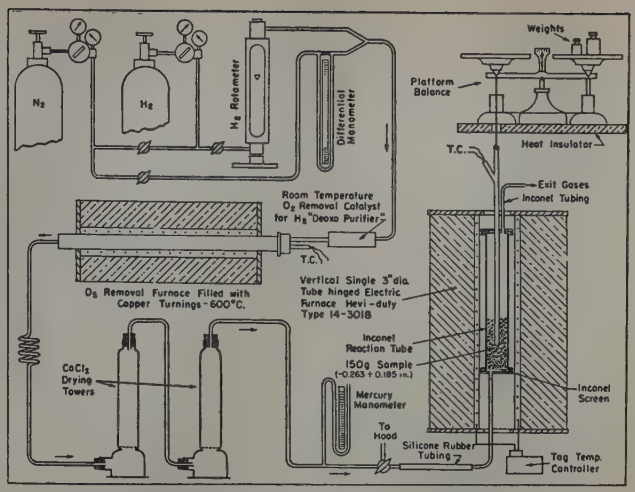


Fig. 2—A diagram of the hydrogen reducibility apparatus. The reducibility test was conducted at 800°C for 90 min, and loss in weight measurements were taken at intervals of 5 to 10 min. Temperature in the reaction chamber was then raised to 1000°C and reduction continued for 1 hr. Throughout reduction hydrogen flow rate was fixed at 0.124 cfm.

U. S. Steel Corp.[10] This test was originally developed in South Kensington, England, at the Imperial College of Science and Technology.[11] The test apparatus, illustrated in Fig. 3, measures the following properties of sinters: 1—Bulk density, grams per cubic

Table IV. Surface Area Measurements of Plant Sinters by the B.E.T. Method, Liquid Nitrogen Adsorption

Sinter	Specific Surface, Sq Cm Per G	Comparative Order* Specific Surface	True Density, G Per CC	Comparative Order* True Density
A	3500	1	4.17	7
B	250	10	4.36	6
C	2000	5	4.17	7
D	2400	4	4.14	7
E	700	8	3.91	9
F	2900	2	4.17	7
G	600	9	4.53	4
H	770	8	4.11	7
I	3300	1	4.29	6
J	100	10	3.70	10
L_1	500	9	5.09	1
L_2	530	9	5.02	1
M	300	10	4.60	4
N	80	10	4.60	4
O	790	8	4.28	6
P	1400	6	4.16	7
Q	1200	7	4.40	5
Open hearth	930	8	4.74	3
Swedish sinter	200	10	4.72	3
Group 3 fines ore	89000	Very high	4.35	6

* High value, 1; low value, 10.

centimeters. 2—Total porosity, percent, volume occupied by helium. 3—Macro porosity, percent, volume occupied by mercury at atmospheric pressure which penetrates pores in excess of +14 micron size. 4—Micro porosity, percent and cubic centimeters per gram, difference in volume occupied by helium and mercury at atmospheric pressure. This value is a measure of the −14 micron pores. 5—Particle density, grams per cubic centimeter, substantially identical to the true density obtained by the B.E.T. surface area measurement.

Table V summarizes results of the S.K. porosity test on the plant survey sinter samples. Values are discussed in a later section of this paper.

Davis Tube Magnetic Test: It has been felt by many investigators that the degree of oxidation is an important factor in determining strength and reducibility of sinters. An evaluation was made of a

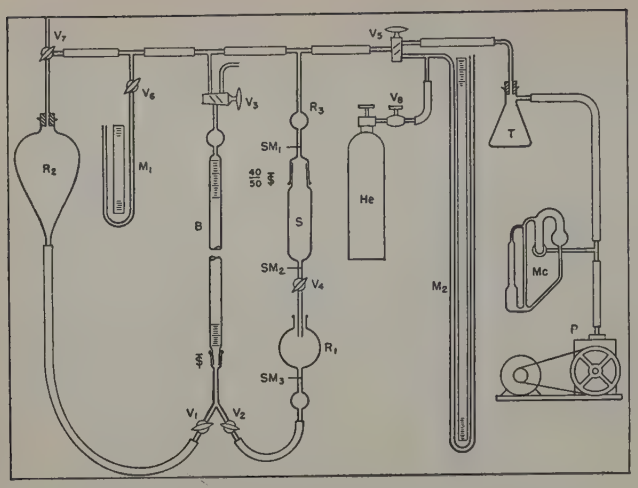


Fig. 3—The S.K. porosity testing apparatus shown above measures bulk density of sinters; total porosity, percent; macro porosity, percent; micro porosity, percent and cubic centimeters per gram; and particle density, grams per cubic centimeter.

Davis tube magnetic tester which magnetically separates the magnetic particles in a −100 mesh sample. The sinter with the highest percentage of magnetic material as given in Table VI was sample M with 94.3 pct; lowest values were obtained with sinter C, 65.6 pct, and Swedish sinter, 67.0 pct. The percentage of magnetic fraction consists not only of magnetite but other magnetic fractions as well, including some physically bound pieces of non-magnetic material. Magnetic measurements were not made on group 3 fines ore.

X-ray Diffraction Analysis: X-ray diffraction analyses of all the plant survey sinters were made to determine relative amounts of magnetite and hematite in each sample. Table VI gives the results obtained by X-ray diffraction. Prior to obtaining these values, the X-ray crystallographers prepared a curve of percentages of hematite and magnetite vs relative line intensities using physical mixtures of cp-grade hematite and magnetite. The X-ray technique used consisted of determining the relative intensities of Fe_2O_3 and Fe_3O_4 lines on a Debye-Scherer photographic negative obtained when the samples were radiated with Fe K α radiations. Therefore results in Table VI are actually iron oxide distributions. In assessing the degree of oxidation of magnetite to hematite, relative amounts of each in the total iron oxide content are of interest. Seventy percent of the iron oxide in Swedish sinter occurs in the form of hematite, an extremely high percentage. The highest percentage of iron oxide as hema-

Table V. S. K. Porosity Test of Plant Sinter Using Mercury and Helium

Sinter	Bulk Density, G Per CC	Comparative Order*	Total Porosity, Pct	Comparative Order*	Macro Porosity, Pct†	Comparative Order*	Micro-Porosity‡ Pct	Micro-Porosity‡ Comparative Order*	Particle Density, G Per CC
A	1.49	4	63.8	8	58.8	8	5.0	2	4.12
B	1.40	5	67.0	6	63.8	6	3.2	6	4.22
C	1.36	6	67.9	6	62.4	6	5.5	2	4.26
D	1.50	4	64.5	8	60.5	7	4.0	4	4.20
E	1.68	1	59.6	10	56.6	9	3.0	6	4.15
F	1.29	7	68.9	5	65.4	5	3.5	5	4.20
G	1.55	3	65.9	7	62.3	6	3.6	5	4.55
H	1.40	5	66.7	6	61.2	7	5.5	2	4.14
I	1.56	3	63.3	8	58.4	8	4.9	3	4.23
J	1.12	10	69.3	5	66.8	4	2.5	7	3.62
L_1	1.22	8	75.9	1	72.9	1	3.0	6	5.08
L_2	1.17	9	76.6	1	74.0	1	2.6	7	5.00
M	1.52	3	66.9	6	62.9	6	4.0	4	4.58
N	1.50	4	67.7	6	63.6	6	4.1	4	4.64
O	1.49	4	66.3	7	64.7	5	1.6	9	4.42
P	1.28	7	68.8	5	64.6	5	4.2	4	4.10
Q	1.35	6	68.7	5	65.8	5	3.0	7	4.33
Open hearth	1.70	1	64.1	8	59.4	8	4.7	3	4.73
Swedish sinter	1.32	7	71.9	3	70.8	2	1.1	10	4.70
Group 3 fines ore	1.43	5	66.2	7	53.2	10	13.0	1	4.23

* High value, 1; low value, 10.
† +14 micron.
‡ −14 micron.

Table VI. Relative Amounts of Magnetite and Hematite in Plant Sinters

Sinter	Davis Tube, Pct Magnetic	Comparative Order,* Pct Magnetic	X-Ray Diffraction: $\frac{Fe_3O_4}{Fe_2O_3 + Fe_3O_4} \times 100$	X-Ray Diffraction: Ratio of Magnetite to Hematite, Pct	X-Ray Diffraction: Other (Probable) Compounds	X-Ray Diffraction: Comparative Order,* Ratio of Magnetite to Hematite, Pct
A	76.5	7	79	3.76		7
B	83.0	4	86	6.14		4
C	65.6	10	70	2.33	Few pct quartz	8
D	74.3	7	77	3.35	Few pct quartz	7
E	85.0	4	86	6.14		4
F	81.0	5	84	5.25	5 pct Fe, 5 pct FeO	5
G	80.2	5	82	4.56		6
H	71.9	8	76	3.17	Few pct quartz	7
I	71.7	8	80	4.00		6
J	75.1	7	85	5.67	Few pct quartz	4
L_1	84.3	4	80	4.00		6
L_2	83.8	4	75	3.00		7
M	94.3	1	90	9.00		1
N	74.8	7	83	4.88		5
O	88.9	2	85	5.67		4
P	74.1	7	74	2.85		8
Q	84.9	4	79	3.76		7
Open hearth	93.8	1	88	7.33		2
Swedish sinter	67.0	10	30	0.43		10

* High value, 1; low value, 10.

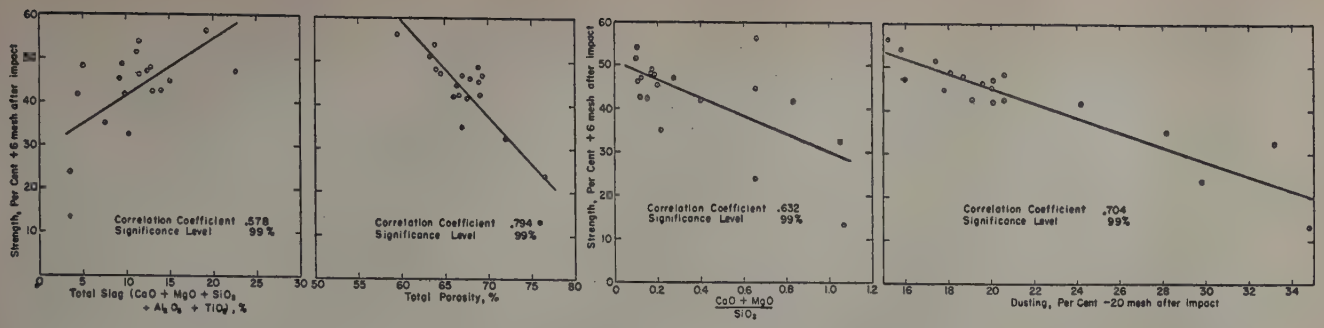

Strength, Per Cent +6 mesh after impact
Correlation Coefficient .578
Significance Level 99%
Total Slag (CaO + MgO + SiO2 + Al2O3 + TiO2), %
Correlation Coefficient .794
Significance Level 99%
Total Porosity, %
Correlation Coefficient .632
Significance Level 99%
CaO + MgO / SiO2
Correlation Coefficient .704
Significance Level 99%
Dusting, Per Cent -20 mesh after impact

Fig. 4—Strength varies directly with total slag and inversely with slag ratio $\frac{CaO + MgO}{SiO_2}$, total porosity, and dusting.

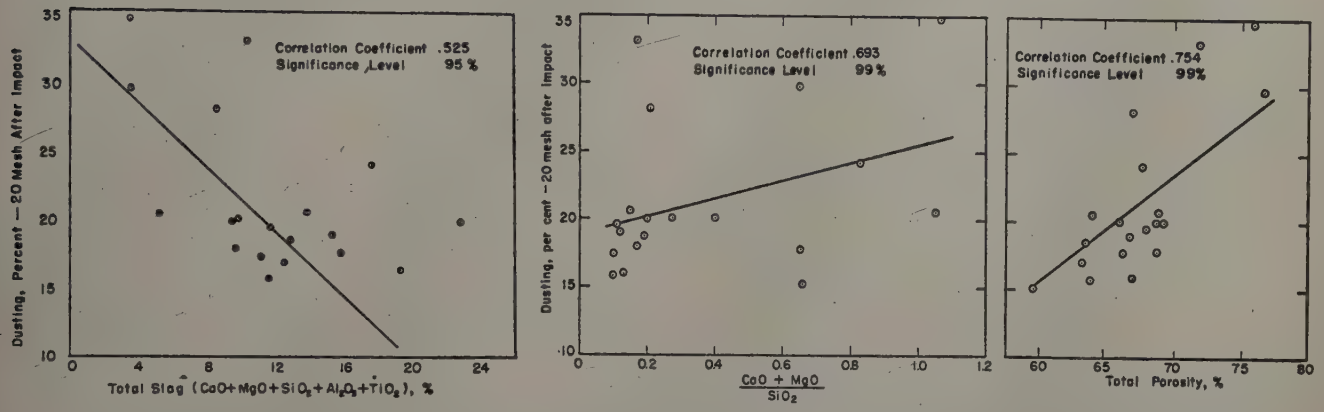

Dusting, Percent — 20 Mesh After Impact
Correlation Coefficient .525
Significance Level 95%
Total Slag (CaO+MgO+SiO2+Al2O3+TiO2), %
Dusting, per cent - 20 mesh after impact
Correlation Coefficient .693
Significance Level 99%
CaO + MgO / SiO2
Correlation Coefficient .754
Significance Level 99%
Total Porosity, %

Fig. 5—Dusting varies directly with slag ratio and total porosity but varies inversely with total slag. Relative reducibility, microporosity, FeO, and Fe_3O_4 are not related to strength and dusting.

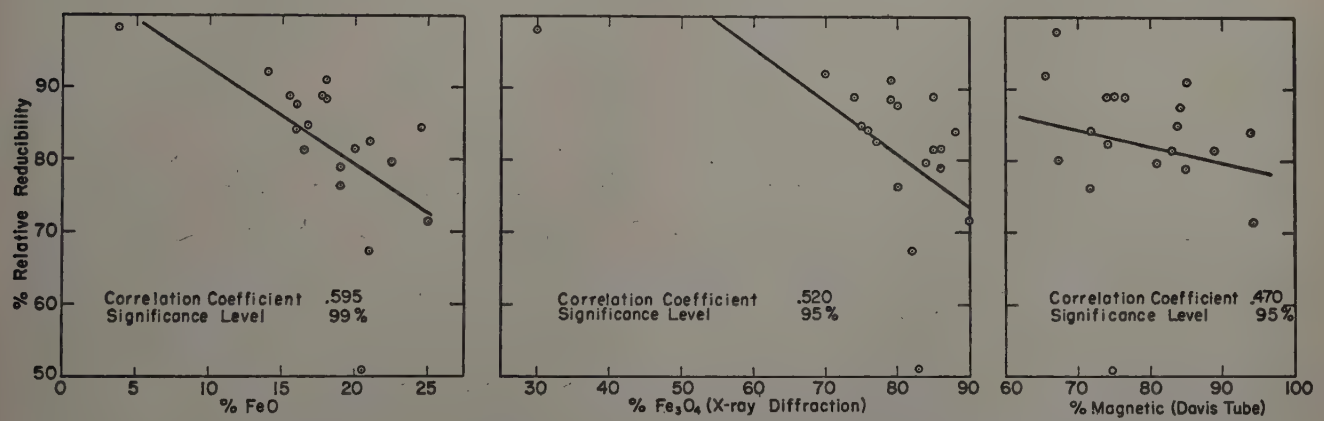


Fig. 6—Relative reducibility is inversely proportional to the Davis tube percentage magnetic, FeO, and Fe_3O_4 (X-ray), but it is not related to the slag ratio, total slag, strength or dusting. Relative reducibility is related directly to surface area. All measurements of the degree of oxidation of magnetite to hematite are directly proportional to each other, namely, FeO, Fe_3O_4 (X-ray), and Davis tube percentage magnetic.

tite in American sinter was 30 pct in sample C, the lowest 10 pct in sample M.

Microstructure: The study of the microstructure of sinters has been undertaken to identify constituents, determine the oxidation pattern, and formulate bonding theories. Although microstructure studies have been of great value, it was very difficult to arrive at a definite average structure for the sinter sample of each plant, since sinters are extremely heterogeneous.

Slag Relationships in Sinters: Table I indicates the slag ratio $\frac{CaO + MgO}{SiO_2}$ and total slag ($CaO + MgO + SiO_2 + Al_2O_3 + TiO_2$) of the plant sinter samples. Although FeO might have played an important role in affecting slag properties, its accurate determination in slag content of the sinters was an insurmountable problem at the time. According to Table VI only sinter sample F contained sufficient FeO to be detected by X-ray diffraction. None of the other samples contained sufficient FeO or slag phases containing FeO to be indicated by X-ray diffraction. The FeO, therefore, was not considered in these slag relationships. The range of the slag ratio obtained in the plant sinter samples was 0.099 to 1.065. Range in total slag was 3.47 to 22.60 pct.

Correlation of Test Data: Since many of the tests on plant sinters were believed to be measuring related properties as well as identical properties in the case of X-ray analysis and chemical analysis for FeO,

Table VII. Correlation of Sinter Properties

Property	Correlatable Property	Correlation Coefficient	Confidence, Pct	Proportionality Relationship	Non-Correlatable Property
Strength	Total porosity	0.794	99.0	Inversely	Microporosity
	$\frac{CaO + MgO}{SiO_2}$	0.632	99.0	Inversely	FeO, pct
	Total slag	0.578	99.0	Directly	Relative reducibility, pct
	Dusting	0.704	99.0	Inversely	
Dusting	Total porosity	0.754	99.0	Directly	Fe_3O_4 (X-ray), pct
	$\frac{CaO + MgO}{SiO_2}$	0.693	99.0	Directly	Relative reducibility, pct
	Total slag	0.525	95.0	Inversely	
	Strength	0.704	99.0	Inversely	Microporosity
Relative Reducibility, pct	Davis tube, pct Magnetic	0.470	95.0	Inversely	$\frac{CaO + MgO}{SiO_2}$
	FeO, pct	0.595	99.0	Inversely	Total slag
	Fe_3O_4 (X-ray), pct	0.520	95.0	Inversely	Dusting, strength
	Surface area	0.444	90.0	Directly	Total porosity
Fe, pct	Particle density	0.691	99.0	Directly	
FeO, pct	Fe_3O_4 (X-ray), pct	0.887	99.0	Directly	Strength
	Relative reducibility, pct	0.595	99.0	Inversely	
	Davis tube, pct magnetic	0.610	99.0	Directly	
Fe_3O_4 (X-ray), pct	FeO, pct	0.887	99.0	Directly	Dusting
	Davis tube, pct magnetic	0.619	99.0	Directly	Surface area
	Relative reducibility, pct	0.520	95.0	Inversely	
Davis tube, pct magnetic	Fe_3O_4 (X-ray), pct	0.619	99.0	Directly	Surface area
	Relative reducibility, pct	0.470	95.0	Inversely	
	FeO	0.610	99.0	Directly	
Total porosity	Strength	0.794	99.0	Inversely	Surface area
	Dusting	0.754	99.0	Directly	Relative reducibility, pct
	$\frac{CaO + MgO}{SiO_2}$	0.458	95.0	Directly	
	Total slag	0.473	95.0	Inversely	
Microporosity	Surface area	0.503	95.0	Directly	Dusting Strength
Surface area	Microporosity	0.503	95.0	Directly	Total porosity Relative reducibility, pct Fe_3O_4 (X-ray), pct Davis tube, pct magnetic
$\frac{CaO + MgO}{SiO_2}$	Strength	0.632	99.0	Inversely	Relative reducibility, pct
	Dusting	0.693	99.0	Directly	
	Total porosity	0.458	95.0	Directly	
Total slag	Strength	0.578	99.0	Directly	Relative reducibility, pct
	Total porosity	0.473	95.0	Inversely	
	Dusting	0.525	95.0	Inversely	
	Correlation coefficient	0.5751	0.5285	0.4555	0.3887
	Confidence, pct	99	98	95	90

it was decided to calculate correlation coefficients using standard statistical procedures. In these calculations two tests were compared by treating their results to determine significant linear relationships. Only results of the 19 plant sinter samples were used in the statistical calculation, since group 3 fines iron ore was used as a base line, and in many instances its properties were outside the normal spread of values for sinters. Table VII presents the correlation coefficients obtained by use of a formula from Ezekiel.[12] In Table VII each correlation coefficient is further clarified by presentation of the level of significance of the value or the confidence limit.[13] As an example of this significance, if a correlation coefficient has a numerical value equal to or higher than 0.4555 (95 pct significant) but lower than 0.5285 (98 pct significant) for a comparison of two properties, it is 95 pct certain that a relationship exists between the two properties.

To illustrate further the significance of the relationships uncovered by statistics, test data were plotted in Figs. 4, 5, and 6. To determine the *line of best fit* through the plotted data, the *method of least squares*[14] was employed. This straight line was plotted through the data, expressing an empirical relationship where estimates of the dependent variable (ordinate) may be made according to the linear function for any value of the independent variable (abscissa).

Using the statistical procedures outlined, a list of relationships was revealed between pairs of several sinter properties. These relationships, presented in Table VII and Figs. 4, 5, and 6, may be summarized as follows.

1—Strength varies directly with total slag and inversely with slag ratio $\frac{CaO + MgO}{SiO_2}$, total porosity, and dusting. Dusting varies directly with the slag ratio and total porosity but varies inversely with total slag. Relative reducibility, microporosity, FeO and Fe_3O_4 are not related to strength and or to dusting.

2—Relative reducibility is inversely proportional to the Davis tube percentage magnetic, FeO, and Fe_3O_4 (X-ray), but it is not related to the slag ratio, total slag, strength or dusting. Relative reducibility is related directly to surface area. All measurements of the degree of oxidation of magnetite to hematite are directly proportional to each other, namely, FeO, Fe_3O_4 (X-ray), and Davis tube percentage magnetic.

3—The total iron percentage is directly proportional to the particle density.

4—Surface area is directly proportional to microporosity and relative reducibility, but surface area and microporosity are not related to any other property statistically appraised.

Discussion of Results

In a discussion of test results in plant survey sinters, the inadequacy of sampling based on a 400-lb grab sample of sinter at only one period must be taken into account. Furthermore, since there is no assurance that representative samples were secured, no comparisons can be made between the plant sinters. The important results of the experimentation were the development of test methods and the fact that a marked differentiation could be made between sinters of such varying compositions. It is hoped that some plants will be sufficiently interested in results of the tests reported to apply these methods in production trials on their sinters. Such trials, combined with blast-furnace production and operating data, would be of value in establishing requirements for good sinter in the blast-furnace.

From the viewpoint of a sinter plant operator, test results revealing strength, dusting, and relative reducibility would be of primary importance. Fortunately an increase in strength accompanied decrease in dusting; consequently the two can be considered together (from Table VII and Figs. 4, 5, and 6) as being related to total porosity, total slag, and the slag ratio, $\frac{CaO + MgO}{SiO_2}$. This information may be interpreted as follows: the slag ratio and total slag content control the total porosity, which in turn determines strength and dusting. With increasing slag content and decreasing slag ratio (more acid), a low porosity results. With low porosity, the strength is dependent upon a large cross-sectional area available to fracture and is therefore high. With high porosity resulting from an increasing slag ratio (approaching neutral) and a decreasing slag content, the cross-sectional area available to fracture is small and the strength is low. In summary, a high strength is obtained with highly acid slags and sufficient slag content to coat over the particles, thereby causing low porosity. Therefore, if this interpretation is correct, the sinter plant operator may control strength by controlling the slagging constituents.

In considering reducibility of the sinter, the sinter plant operator would be interested in the degree of oxidation of magnetite to hematite. With increasing magnetite in the sinters, reducibility was decreased; with increasing hematite content in the sinters reducibility was increased. The outstanding example of this is the Swedish sinter sample, which had a very high hematite content and resultant high reducibility. However, one fact is thought-provoking, namely, the lack of correlation between strength and reducibility or strength and degree of oxidation. Other investigators[15] have indicated correlations between strength and reducibility. Perhaps the fact that sinter samples used for this evaluation were produced under widely divergent conditions and from a variety of raw materials explains this lack of correlation between strength and reducibility.

Conclusions

Nineteen sinter samples and one iron ore sample have provided a broad evaluation of test methods used to determine chemical and physical properties. The following factors were determined from the evaluation.

1—Impact test results of strength and dusting were related to total porosity, which was explained in terms of a slag ratio $\frac{CaO + MgO}{SiO_2}$ and the total slag content. With highly acid slag and high total slag, strength of sinter produced was high.

2—Relative reducibility test results were influenced by the degree of oxidation of sinter. The chemically analyzed FeO percentage provided the most significant correlative test.

Acknowledgment

Thanks are extended to all the companies and their blast-furnace personnel for supplying production data and sinter samples for this evaluation. Thanks are also due the American Iron & Steel Institute for sponsoring this research at Mellon Institute and for permission to publish the results. The AISI Subcommittee on Agglomeration of Fines, under its chairman, E. G. Hill, greatly assisted in organizing the plant survey in 1949 and has been generous of time and advice while the tests were being developed and data collected.

References

[1] Robert E. Powers: A Survey of Blast-Furnace Sintering Practice. AISI Technical Committee Activities (1950) pp. 185-216; *Steel* (Feb. 5, 1951) pp. 95-108.

[2] Robert E. Powers: Improving Current Practice in Blast-Furnace Sintering. AISI *Yearbook* (1951) pp. 242-270.

[3] E. G. Hill and R. E. Powers: Testing of Sinter. AISI *Yearbook* (1951) pp. 271-288.

[4] U. S. Steel Corp.: *Methods of the Chemists of the U. S. Steel Corporation for the Sampling and Analysis of Iron and Manganese Ores* (1926) pp. 55-60.

[5] F. M. Hamilton and H. E. Ameen: Laboratory Studies on Iron Ore Sintering and Testing. *Trans.* AIME (1950) **187**, pp. 1275-1282.

[6] F. M. Hamilton: Physical Tests and Results on Some Agglomerated Iron Ores. *Proceedings,* Blast Furnace, Coke Oven, and Raw Materials Committee, AIME (1951) **10**, pp. 135-140.

[7] E. P. Barrett and C. E. Wood: Determining Rates of Deoxidation of Iron Oxide Materials in Reducing Gases. Loss-in-weight Method. *Industrial Engineering Chemistry,* Anal. Ed. (1946) **18**, pp. 285-286.

[8] E. P. Barrett and C. E. Wood: Relative Reducibility of Some Iron Oxide Materials. U. S. Bur. Mines *R.I. 4569* (1949).

[9] S. Brunauer, H. Emmett, and E. Teller: Adsorption of Gases in Multimolecular Layers. *Journal Amer. Chem. Soc.* (1938) **60**, pp. 307-319.

[10] R. J. Morton: Testing of Iron Ores and Agglomerates Related to Blast-furnace Use. *Proceedings,* Blast Furnace, Coke Oven and Raw Materials Committee, AIME (1951) **10**, pp. 122-134.

[11] H. L. Saunders and H. J. Tress: Ore Testing, the "S.K." Porosity Test. *Iron and Steel* (London, 1945) **18**, pp. 565-567.

[12] Mordecai Ezekiel: *Methods of Correlation Analysis,* pp. 146-152. John Wiley & Sons, Inc., New York, 1947.

[13] R. A. Fisher: *Statistical Methods for Research Workers,* p. 204. Oliver & Boyd, Ltd., Edinburgh, 1944.

[14] Ref. 12, pp. 59-74.

[15] E. C. Rudolphy and D. J. Carney: Quality Control of Blast Furnace Flue-dust Sinter. Blast Furnace, Coke Oven and Raw Materials Conference, Buffalo, N. Y., April 20-22, 1953.

A Guide to the Proper Application of Classifiers

by H. W. Hitzrot

SEPARATING a mixture of particle sizes of material suspended in a liquid medium is by no means an exact science. Selecting machines for individual classifying operations is even more difficult. The plant operator's own background is of course invaluable, and considerable help may be obtained from technical articles, talks with sales engineers, and handbooks on ore dressing. These several sources of information, however, are difficult to marshal in proper perspective for the major decision on classification units that an operator may be called upon to make. To the present writer's knowledge this assembly of facts is not available in handbooks, and technical papers are scarce on classification equipment developed in the past five or six years. It is believed that this paper will be helpful to users of classification equipment at this particular period in the development of hydro-classification.

For easy reference the following classification units now available and in use in metallurgical and industrial operations are listed below, with a brief description of each.

Unit-type classifiers, bowl classifiers, and *bowl desiltors* are all rectangular tanks, slightly tilted, with reciprocating rakes or screws to remove settled sands. The unit-type classifier, Figs. 1 and 2, is available in widths from 14 in. to 20 ft and lengths up to 40 ft. The shallow bowl of the bowl classifier, Fig. 3, equipped with rotating rakes, is superimposed on the lower end of the tank. Reciprocating rake compartments for this design range from 18 in. to 20 ft wide. Bowl diameters vary from 4 to 28 ft. The flat-bottomed bowl of the desiltor, Fig. 4, of relatively large diameter, is equipped with rakes rotating outward and partly over a pit, which is created by extension of the rectangular tank into and under the bowl section. Bowl desiltors are available with reciprocating rake sections 4 to 20 ft wide and bowls from 20 to 50 ft in diam. The bowl desiltor is used for applications beyond the range of the bowl classifier.

The *hydroseparator,* Figs. 5 and 6, is a circular tank equipped with slowly rotating rake arms, set on a slope, with interrupted rake or spiral blades to move the settled solids to a central discharge cone. Tank diameters vary from 4 to 250 ft. Tank depths at center are 2 to 3 ft for small units and up to 25 ft for larger units.

Hydraulic classifiers of the sizer and super-sorter types, Figs. 7 and 8, are narrow, deep, rectangular tanks divided by vertical baffles into a series of pockets. Hydraulic water is added near the bottom of each pocket. Perforated constriction plates, spiral flow arrangements, or jets are used to disseminate the water under pressure (hydraulic water) throughout the bed of material in the pocket. Discharge valves on each pocket are operated automatically by a pneumatic mechanism, a pincer-type mechanism, or a pressure control and motor combination actuated by a hydrostatic tube within the pocket. Hydraulic classifiers are available in 4, 5, 6, and 8-pocket units of varying constriction plate areas to suit conditions. There is now a jet sizer of unit pocket design that can be made up in 1 to 25 sections or more to accommodate sizing requirements.

The *hydroscillator,* Fig. 9, is a rectangular tank set on a slope of 3 to 4 in. per ft. A bowl is superimposed on the lower end. The bowl bottom is an oscillating rubber-covered disk, perforated to allow hydraulic water introduced beneath the disk to set up a teeter bed and thus produce an oversize or rake product exceptionally free of slimes, and material minus the mesh of separation.

A shallow dam at the periphery of the bowl allows the coarse or oversize fraction to spill over and drop down into the tank compartment, where it is moved up the deck by reciprocating rakes. The material, minus the mesh of separation, overflows a circular and stationary weir which is several inches higher than the dam on the oscillating disk. It is carried off in a circular launder in the usual manner.

These units are available in bowl diameters from 4 to 14 ft and with reciprocating rake compartment widths to suit the tons per hour to be handled.

Centrifugal classifiers include the solid bowl centrifuge and the cyclone classifier. The *solid bowl centrifuge,* Fig. 10, consists of a truncated cone fixed to a horizontal shaft and rotating at high speed. An internal spiral rotating at slightly less speed continuously removes solids deposited on the inner surface of the cone, or bowl.

Feed enters the cone by means of the hollow center shaft. Overflow leaves through ports at the large end of the cone, and oversize solids, moved by the spiral, exit through ports at the small end.

Centrifugal classifiers of this type are available in cone diameters from 18 to 54 in.

The *cyclone classifier,* Fig. 11, is a stationary cone having a cylindrical upper section and a lower cone section. Feed is introduced tangentially into the upper cylindrical section under pressure from a hydrostatic head or by means of a pump. Centrifugal force thus induced effects a classification within the cone, the fine sizes being carried off in the overflow through an opening at the top of the cylindrical section. Coarse solids at relatively high pulp density exit through a control valve at the apex of the lower cone section. Cyclone classifiers are available in 3, 6, 12, 14, 24, and 30-in. diam.

The *cone classifier,* Fig. 12, is a steel-plate cone with sides usually about 60° from horizontal. It contains no rotating mechanism. Feed enters a feedwell at center and classification is effected by gravity and pulp density. Fines are carried off in the flow over a peripheral weir at the top of the cone. Settled solids exit through an opening at the apex. An apex valve actuated through levers and rods by the pulp density in the lower cone section is usually supplied. Diameters are usually maximum at 8 ft.

H. W. HITZROT, Member AIME, is Special Assistant for Engineering Sales, the Dorr Co., Stamford, Conn.

Discussion on this paper, TP 3691B, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, June 30, 1952. Mexico City Meeting, Oct. 29 to Nov. 3, 1951.

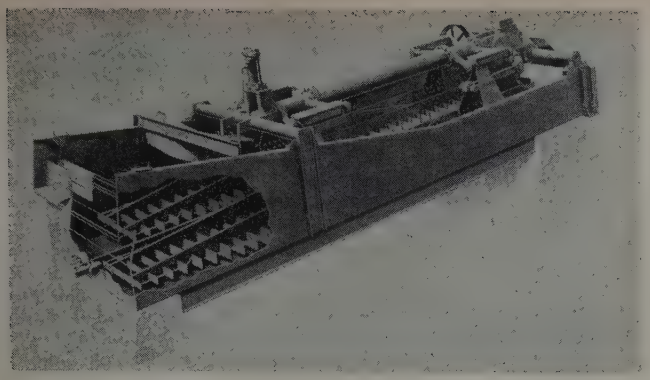


Fig. 1—A unit-type classifier utilizing reciprocating rakes.

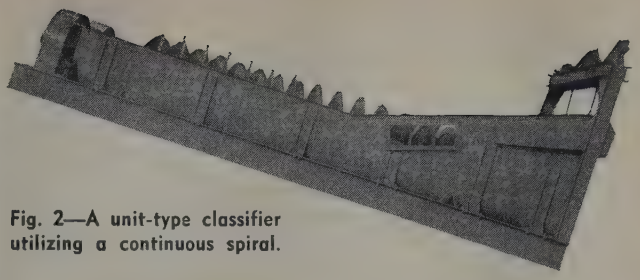


Fig. 2—A unit-type classifier utilizing a continuous spiral.

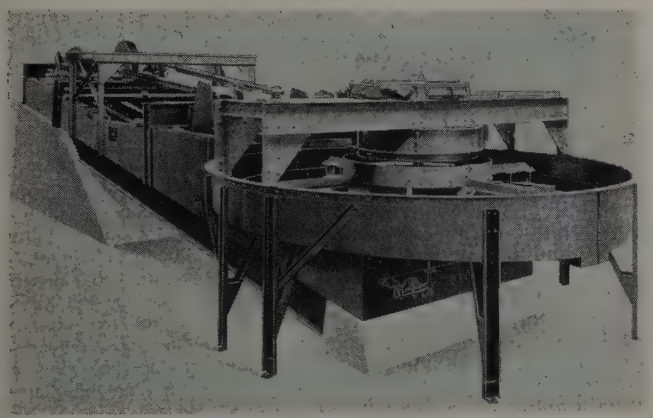


Fig. 3—The bowl-type classifier.

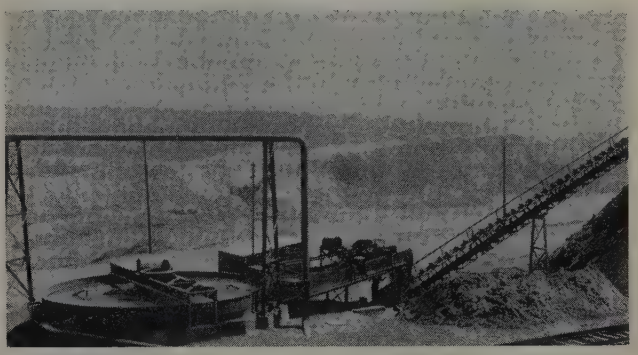


Fig. 4—A typical bowl desiltor installation.

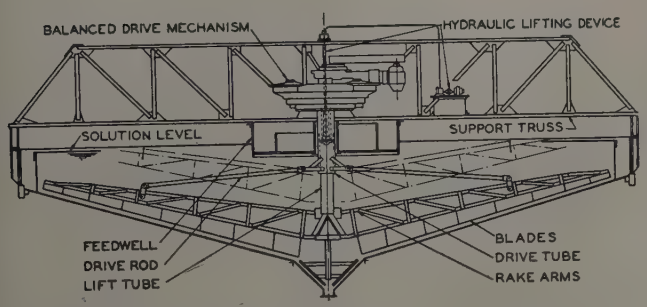


Fig. 5—Illustrative sectional elevation of hydroseparator utilizing interrupted rake blades.

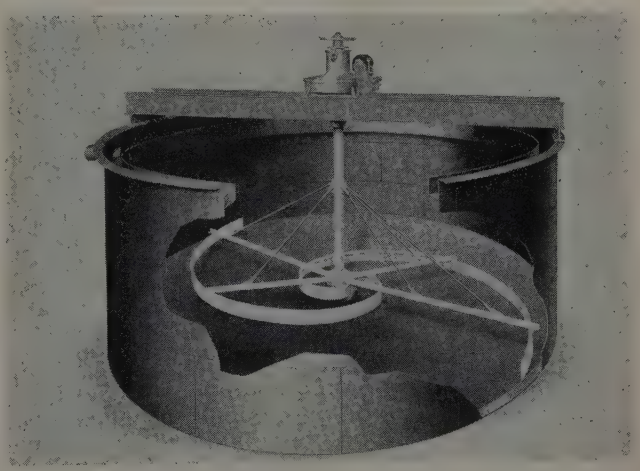


Fig. 6—The hydroseparator, showing spiral blade construction.

Fig. 7—Installation view of a sizer hydraulic classifier.

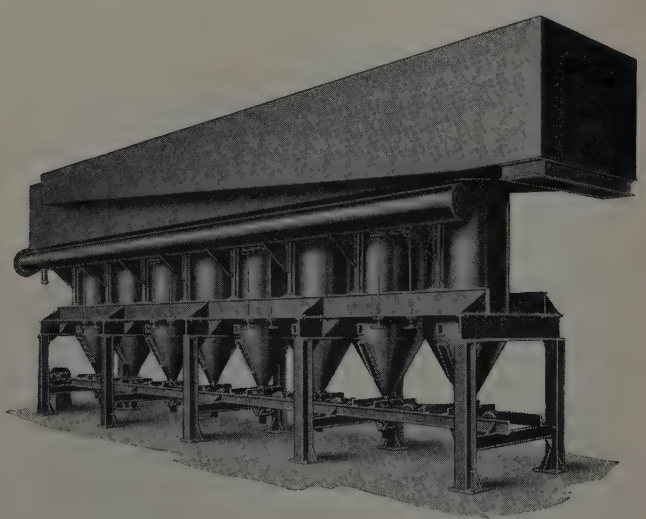


Fig. 8—A super sorter hydraulic classifier.

In the *sand washer,* Fig. 13, a set of buckets is attached to a circular frame rotated slowly on an inclined deck, which forms the bottom of a circular tank. The top of the tank side extending up from the heel becomes the overflow weir. Feed under pressure is introduced toward the center of the inclined bottom. Sand washers are available in 7, 9, and 12-ft diam.

Below what mesh of separation are classifiers more efficient than vibrating screens? Allowing for some latitude of opinion, 20 mesh is about the limit for a two-product separation on a mixed-size material of 2.65 sp gr. For separations coarser than 20 mesh, for example, 10 or 14 mesh, screens are preferable. On mixed-size materials having greater specific gravities, in the range of 3.5 to 5.0, classifiers should be limited to a separation at 28 to 35 mesh. Screens will be more efficient and should be used on two-product separations for 20 mesh and coarser.

Particles coarser than 20 mesh can be made to report in the overflow from unit-type classifiers when the ground ore or material contains a high percentage of true slimes. Such materials are cement rock and ores containing talc and clay. But these coarse particles, sometimes as large as ¼ in., are more in the nature of tramp oversize carried in suspension in a high-density classifier pool. The basic mesh of separation is more nearly at 20 mesh.

In hydraulic classifiers of the *sizer* and *supersorter* types it is possible to make a split at 6 or 8 mesh, but this is done at the expense of about 4 tons of hydraulic water per ton of feed handled and generally would not be practicable for big tonnage operation.

So much for the limitations of mechanical-hydraulic and centrifugal classifiers in general. For each of the nine types of classifiers described in this paper there are individual limits of application, as shown in Table I.

Mechanical-hydraulic classifiers, equipped with either reciprocating or spiral rakes, are the most widely used and have countless applications.

Unit-Type Classifiers

Limitations: Unit-type classifiers are commonly employed for two-product separations, overflow and rake product, with overflow solids ranging between 20 and 100 mesh. Maximum size of feed particle is normally 1 in., occasionally 1½ to 2 in. When specific gravity of the feed solids is 4.0 to 5.0, overflow should be limited to a top size of 28 to 35 mesh.

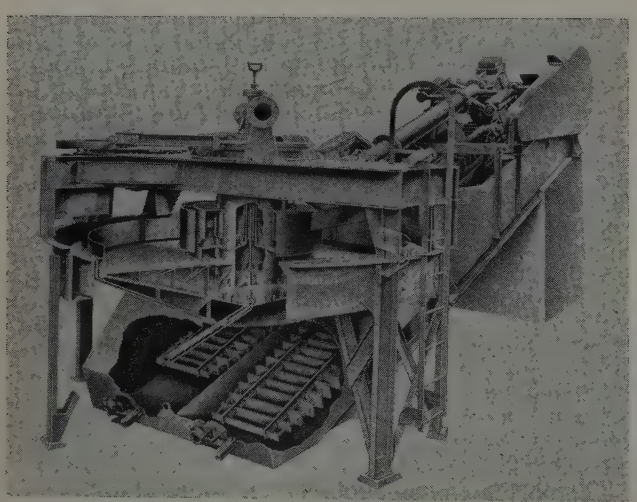


Fig. 9—A cutaway view of the hydroscillator.

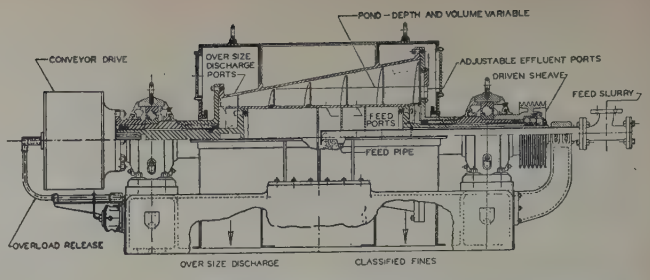


Fig. 10 (above)—A sectional elevation of the solid bowl centrifuge.

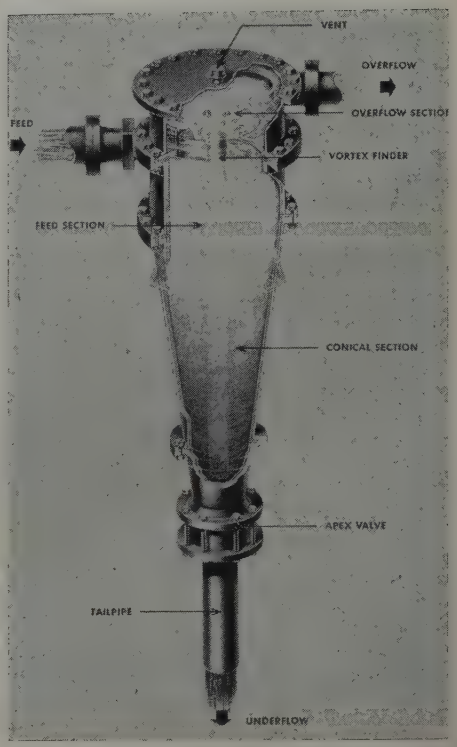


Fig. 11 (right)—Cutaway view of a cyclone classifier.

Control of water into these classifiers is very important, as separation at whatever mesh, 35, 48, 65, or 100, is made largely by the buoyancy or viscosity of the mixture of water and solids in the pool. Agitation of the sand-raking mechanism also affects mesh of separation. Excess water, contrary to the opinion of the uninitiated, ruins the separation by allowing too many particles of the desired size to drop down into the rakes or screws. Too little water, conversely, allows oversize particles to remain in suspension and contaminate the overflow.

When unit-type classifiers are used for scrubbing and dewatering, however, the amount of water entering with the feed is not critical. In most cases the products handled are preponderantly granular and hence fast-settling in character, for example, concentrates and silica sand. The pool area then determines the amount of water that can be handled. Fresh water in the form of wash water is frequently added up on the drainage deck section further to clean the rake product of contaminating slimes.

In both reciprocating and screw types, this classifier produces a rake product of consistent moisture content hour in and hour out, even with some variations in the feed volume. On dewatering operations carried out before further processing this is of great advantage. For instance, in the dewatering of phosphate rock prior to oiling ahead of tabling or prior to the addition of flotation reagents, the content of consistent low moisture solids in the rake product is highly valued.

Applications: Unit-type classifiers are used most frequently with mills for closed-circuit grinding of

Table I. Sizes, Limitations, and Major Applications of Unit Classifiers, Bowl Classifiers, Bowl Desiltors, Hydroseparators, Hydraulic Classifiers, Hydroscillators, Centrifugal Classifiers, Cone Classifiers, and Sand Washers

Type of Classifier	Normal Size Range			Mesh of Separation, Ranges	Relative Classifier Efficiency	Normal Feed, Tonnage Range	Max. Oversize Handled	Normal Overflow, Pct Solids, Range	Normal Feed Density Range, Pct	Normal Underflow or Rake Product, Pct Solids	Motor Range, HP	Hydraulic Water Required	Typical Applications
	Width	Diam	Max. Length										
Unit Classifier	14 in. to 20 ft	—	40 ft	20 to 100	Medium	1 to 350 tph	1 to 1½ in.	5 to 65	Not critical	80 to 83	½ to 25	Spray wash optional	Closed circuit grinding Washing and dewatering Process feed control
Bowl classifier	18 in. to 20 ft	4 to 28 ft	38 ft	65 to 325	Medium plus	1 to 300 tph	½ to ¾ in.	5 to 25	Between 10 and 75	75 to 83	Bowl: 1 to 7½ Recip. rakes: 1 to 25	Water added with feed or in classifier for correct overflow density	Closed circuit grinding, usually in secondary circuit but sometimes in primary. Washing and dewatering
Bowl desiltor	4 to 16 ft	20 to 50 ft	38 ft	100 to 325	Medium	5 to 250 tph	½ in.	1 to 15	Not critical	75 to 83	Bowl: 1 to 10 Recip. rakes: 5 to 25	Spray wash optional	Recovery of fine sand, limestone, coal and fine phosphate rock from large flow volumes.
Hydroseparator	—	4 to 250 ft	—	Usually 100 to 325. Special cases 35 to 325	Low	Few lb per hr to 500 to 700 tph	¼ in.	1 to 20	Not critical Generally 5 to 20	30 to 50	Fractional to 15	All water enters with feed	For fine separation where large feed volumes are involved
Hydraulic classifiers Sizer	Varies with no. of pockets	—	Varies with no. of pockets	6 to 150	High	2 to 100 tph	⅛ to 3/16 in.	5 to 20	40 to 60	40 to 60	1 to 2 for air pressure	Up to 4 tph feed	Gravity concentration of metallic ores Preparation of table feed Sizing of homogeneous materials
Super sorter	6 ft	—	About 40 ft	¼ to 150 ¼ to 150	High High	40 to 150 tph	¼ to ⅜ in.	5 to 20	30 to 60	40 to 60	1 to operate pincer valves	Up to 4 tph feed	Gravity concentration — preparation of table feed —coal Sizing of homogeneous materials
Hydroscillator	4 to 12 ft	4 to 14 ft	30 ft	20 to 200	High	5 to 250 tph	½ to 1 in.	15 to 30	40 to 80	75 to 83	Bowl: 3 to 10 Rakes: 5 to 20	Up to 1.5 tph per ton of feed	Used where exceptionally clean rake sands are needed Closed circuit grinding
Centrifugal classifiers Cyclone	—	3 to 30 in.	9 ft	100 mesh to 5 microns	Medium	10 to 1500 gpm	14 to 20 mesh	5 to 30	1 to 30	55 to 70	Power for pressure head	None	For fine separations in variety fields
Solid bowl centrifuge	—	18 to 54 in.	70 in.	100 mesh to 5 microns	Medium plus	Up to 500 gpm	¼ in.	5 to 30	1 to 30	40 to 70	10 to 50	None	For fine size fractionating down to 5 microns
Cone classifier	—	2 to 12 ft	—	28 to 325	Low	Up to 100 tph	¼ in.	5 to 30	Not critical	35 to 60	None	None	For desliming and primary dewatering
Sand washer	—	7 ft 9 ft 12 ft	—	28 to 65	Medium	25 to 125 tph	1 in.	5 to 20	About 30 to 35	80 to 83	5 to 10	None	For desliming and dewatering large tonnages of bulk material

nonferrous and ferrous ores, cement rock, silica, and pigments. In fine grinding, straight classifiers are often used for single-stage grinding and as the primaries in two-stage grinding. Not infrequently they function as classification units in the secondary stage of two-stage grinding. As would be expected, the width of the unit to be selected depends on the 24-hr tonnage handled, the mesh of separation, and the specific gravity of the material.

Unit-type classifiers are also employed for final scrubbing, washing, and dewatering of granular products such as phosphate rock, ground silica, concrete sand, fine coal, limestone, precipitated alumina, and mineral concentrates from flotation and gravity concentration milling. Occasionally these classifiers size, wash, and dewater granular organic-chemical products in plastics and related fields.

Reciprocating rake-type units have wide application as grit-removal devices on *detritors,* which remove coarse solids in municipal sewage-disposal plants. They also function as grit and unburned core removal devices in continuous slaking of burned lime. These combination units are known as *slakers.*

Another application of reciprocating rake units is the washing of coarse ore: gold ores, bauxite, iron ores, and marls in cement-manufacturing processes. These units, called *washers,* are reciprocating rake classifiers with separately driven wire mesh or punched plate trommels superimposed over the pool end and partly submerged in it.

A machine of this type can produce three products: a washed coarse material about —3 in. from the trommel, a —⅜ or ½-in. rake product, and finally an overflow —48, 65, or 100 mesh to dust, all of which is carried off with the overflow water.

Employment of unit-type classifiers in the field of ore concentration by the heavy-media process is most worthy of mention. They are used as separatory vessels for both coarse and fine ore fractions. The screw or spiral-equipped unit is particularly applicable as a densifier unit: the screw can be lowered into a bed of heavy media (finely ground magnetite or ferro-silicon) and stored in the classifier pool. Density in the heavy-media circuit is thus periodically raised.

The Bowl Classifier

Limitations: The bowl classifier has been designed and developed for separations finer than are generally attempted in the straight or unit-type classifiers. For the more usual 2.65 to 3.0 sp gr materials, a two-product separation at 65 mesh is the generally accepted top limit. Separations within the range of 100, 150, 200, and 325 mesh are, however, more common. The raking mechanisms of this unit can handle a feed containing particles as large as ½ to ¾ in. For materials of 3.5 to 5.0 sp gr, the top limit for a two-product separation with the bowl classifier is about 100 mesh, and because of the heavier gravity, fineness of separation can be extended below 325 mesh (43 microns) or down to about 20 microns (750 mesh).

At this time diameter of the bowl section does not exceed 28 ft. This limitation is imposed by design difficulties of balancing and rigidity for the overhanging rake section extending underneath the midpoint of the bowl and about 2 ft beyond it.

As with the unit classifiers, water entering the bowl classifier must be controlled. Whereas unit classifiers generally overflow at 25 to 40 pct solids, bowl classifiers overflow at 5 to 25 pct solids; the

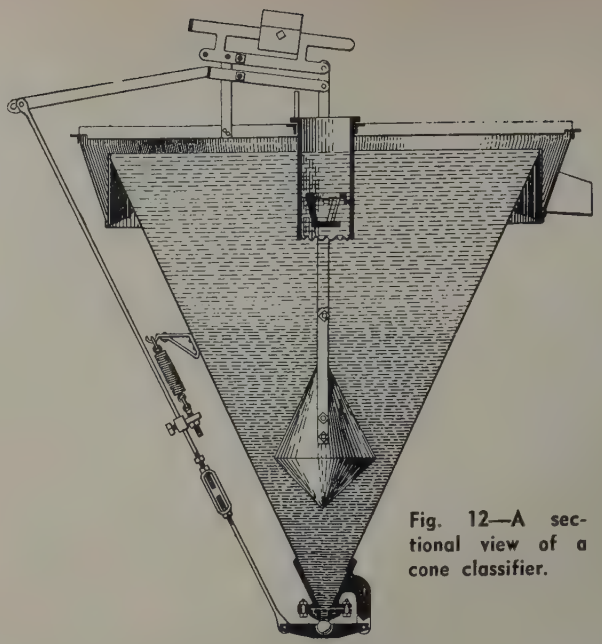


Fig. 12—A sectional view of a cone classifier.

finer the mesh of separation, the lower the percent of solids in both unit and bowl classifiers.

A bowl classifier, when used in closed-circuit grinding, must handle a large tonnage of circulating sands in addition to the new feed. In such cases it is essential to equip the classifier with a reciprocating rake section of a width approximately 80 pct of bowl diameter. Thus for a 20-ft diam bowl the reciprocating rake section should be 16 ft wide, with four rakes 4 ft wide. Reciprocating rakes are standard in 2, 3, and 4-ft widths and an even number of rakes must be used. Hence a 15-ft diam bowl with reciprocating rake section 12 ft wide would have four rakes 3 ft wide.

Applications: The bowl classifier is used most commonly in the secondary or final grinding stage in the closed-circuit grinding of nonferrous and ferrous ores, cement rock and marls, silica sand, and pigments. It is less frequently used in the single-stage fine grinding of these same materials and in the regrind circuits for middlings and concentrates.

The bowl classifier also has wide application as a deslimer. This is by virtue of the fact that any water added into the reciprocating rake compartment has but one means of escape up through the opening in the bowl bottom. Solids collected in the bowl enter the reciprocating rake compartment through this same opening and lose much of their slimes and fines content in the rising stream.

The Multi-Deck Washing Classifier

In some processing operations it becomes desirable to wash a contaminating chemical solution from the rake product of a bowl or a unit-type classifier. In most instances more than one washing stage is required, and one, two, or three additional raking sections are added in cascading fashion to make a unit-type or a bowl classifier with multidecks for the counter-current washing of the sand product. Fresh water is added on the final deck and progresses downward counter-currently to movement of the solids. The wash water flows from one raking section to the next lower section by means of interconnecting launders.

Multi-deck washing classifiers have widest application in the chemical processing field for the counter-current washing of nickel compounds, chro-

mium ores and compounds, alumina, abrasives and abrasive intermediates, tri-sodium phosphate, zeolite sands, and some other chemical compounds. Acid-proof construction must be used for some operations.

The Bowl Desiltor

Limitations: The bowl desiltor is a classification unit of fairly recent design and usage that bears some semblance to the old *Clabowl* used in the china clay and filler clay fields. Its use is limited to operations involving large flow volumes and fine separations where the former goes beyond the range of the maximum diameter (28 ft) of bowl classifiers, or where the relatively small amount of solids in the large volume of flow does not justify the heavy duty construction of bowl classifiers.

The bowl desiltor makes a two-product separation with the mesh of separation in 150 to 200 or 325 mesh range. Usually the tonnage of coarse or rake product is relatively small and more often than not, duplex rakes will handle it. This machine neatly combines in one unit the use of a hydroseparator for the fine separation and a unit-type classifier to dewater the dilute hydroseparator underflow.

The top size for the coarsest particles in the feed to this unit should not exceed ½ in. Certainly there would be few occasions when any solids coarser than about 6 mesh would be in the feed, but the rakes will handle a limited amount of ½-in. pieces.

Applications: At the present time the bowl desiltor has widest application in the recovery of extremely fine sand overflowing the various washing units in concrete sand, foundry sand, glass sand, and limestone-processing plants. Such recovered fine sand is delivered in a drained condition by the reciprocating rakes and is used as a special product or is blended into the coarser product to increase the fineness modulus.

It is conceivable that in the future the bowl desiltor will be used whenever it is desirable to remove a small amount of finely divided solids from a large volume of flow, that is, for the recovery of fine coal, phosphate sand, blast furnace flue dust, or fine mineral concentrates.

The Hydroseparator

Limitations: In the hydroseparator a widening in the line of flow allows the coarser particles to settle out from the suspension. Only the cone classifier is simpler in construction. The hydroseparator is frequently misused because its limitations are not fully recognized.

Classification efficiency of the hydroseparator is appreciably lower than that of the unit-type and bowl classifiers. It depends entirely on gravity settling for the separation, and the overflow can be set to contain around 1 pct or less of particles plus the mesh of separation. This is done at the expense of allowing a relatively high separation of particles minus the mesh of separation to settle into the underflow, resulting in low classification efficiency. The underflow will always contain about 40 to 60 pct of the material minus the mesh of separation, the higher percentage being approached when coarse separations in the 35 to 65 mesh range are adopted.

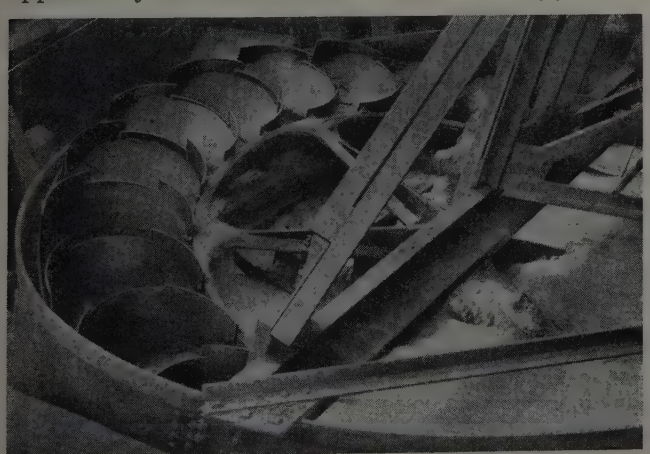


Fig. 13—Installation view of a typical sand washer.

The hydroseparator is generally used for separations finer than 100 mesh, except when the feed flow is so great that the cost of the multiplicity of bowl or unit-type classifiers required makes a coarse separation by means of a hydroseparator attractive. Feed to a hydroseparator should not contain particles much over ¼ in., and when there is an appreciable amount of 28 mesh to ¼-in. material present, as in concrete sand, then heavy duty mechanisms with arms at a slope of 2 to 3 in. per ft should be used.

There is no limitation on the handling of heavy-gravity material in hydroseparators, but the steep rake arm slopes mentioned above should be used.

The size or diameter of a hydroseparator is a function of the feed volume and the separation desired. The tonnage of solids to be handled does not directly affect the size but must be taken into account in selecting the design and height of raking mechanism. The tonnage handled can range from a few pounds up to 500 to 700 tons per hr.

Applications: The comparatively low classification efficiency of the hydroseparator restricts its use to situations where the volume of flow is beyond the range of unit classifiers, bowl classifiers, and the bowl desiltor. It is used, however, for small feed flow volumes where no great importance is attached to the poor classification feature.

The hydroseparator is appropriate when a large volume of water is to be sloughed off prior to further processing of the granular fraction, and units of 75, 100, and 150-ft diam are applicable. Under special conditions units of 200 to 250-ft diam might be used for such problems. Typical applications are the primary hydroseparation of phosphate rock matrix, primary dewatering and scrubbing of concrete sand or other silica sand products following wet screening, and recovery of fine coal in coal washing operations, both in the anthracite and bituminous fields. Further classification or dewatering of the granular solids settled in the hydroseparator is carried out in subsequent operations in all the above cases, so that the limited classification and dilute underflow of the hydroseparator are accepted because of its capacity to handle high flows.

In past years hydroseparators have been used to size abrasive products and to process clays and pigments and a variety of chemical precipitates. In view of the recent development of centrifugal classifiers and the bowl desiltor, it is the author's opinion that hydroseparation problems in the fields mentioned above will be solved in the main by these two types of classifiers.

Hydraulic Classifiers: Sizers and Super Sorters

Limitations: All classifiers thus far discussed except the washer (unit-type) have been two-product machines, coarse and fine. Eight of the nine main types, in fact, are limited to two products.

The sizer and super sorter are multi-pocket units and, therefore, multi-product. Thus for any problem requiring more than two sizes of products from

one machine, the multi-pocket hydraulic classifier is the only one available. It is made up with varying constriction plate areas to meet tonnage requirements, and the number of pockets used depends on the range of products to be made and the weight distribution in the screen analysis of the feed.

Feed to any hydraulic classifier should not contain particles coarser than ¼ in., preferably not over ⅛ or 3/16 in. The classification pockets in series each served by hydraulic water under low pressure will each make a relatively clean size split combining roughly two screen sizes. That is to say, the first pocket on a −14 mesh feed will entrap practically all the +20 and a good part of the +28. The second pocket will entrap the remaining +28 with the bulk of the +35 and some of the +48, and so on down through the several pockets.

The −100, +150 mesh particles are about the finest that can be entrapped and cleaned free of the −150 with any degree of efficiency. Thus all particles −150 mesh will report in the overflow of the sizer and must be further provided for if they are to undergo additional classification or are to be saved.

All hydraulic classifiers operate best when receiving a fairly constant volume of feed somewhere between 40 and 60 pct solids. A unit-type classifier, particularly of the rake type, is ideal for feeding one or more sizers or super sorters.

Another limitation of hydraulic classifiers is the high moisture underflow discharge from each pocket, ranging from 40 to 60 pct solids. Frequently provision must be made for further dewatering.

The super sorter is made up with equal-sized pockets in line with mechanical pincer-type underflow valve controls actuated from a single drive. The latest design of *jet* sizer also is made up of equal-sized pockets, but they can be arranged in line or in pyramid as the underflow valves are actuated by an individual pneumatic control.

Hydraulic classifiers are widely used to prepare sized products for gravity concentration by means of tables or similar devices embodying free settling or hindered settling. This is the major application of sizers and super sorters.

An important field for this type of classifier is the beneficiation of iron, tin, titanium, tungsten, vanadium, chromium, zinc, and lead, and such nonmetallics as coal, phosphate rock, and silica sand. It should be mentioned at this point that the super sorter was especially designed as a high-tonnage unit for use in the cleaning of fine coal.

Another less prominent but increasingly significant field of application for sizers and super sorters is the separation of homogenous products into various size fractions. Natural silica sand is an example. Foundry and glass sands are sold according to grain sizes or grades, and concrete sands frequently require one or more size fractions.

In a few instances the sizer has been used to retreat smokeless powder and to size chemical compounds such as sodium aluminate crystals.

The Hydroscillator

Limitations: Separation with the hydroscillator ranges between 20 and 200 mesh inclusive. That is to say, operating conditions can be set to overflow from the oscillating bowl 20 mesh at the coarse limit and at any mesh on down through 28, 35, etc., to 200. Coarse particles up to ½ or 1 in. in the feed can pass through the oscillator compartment without difficulty.

Hydraulic water at the rate of 0.5 to 1.5 tons per ton of feed is necessary to effect the highly efficient separation. For the required separation with a reasonable amount of hydraulic water, feed should be in the form of a thick slurry for separations in the coarser meshes. For separations in the finer meshes a dilute feed is permissible. The oversize fraction of the feed as delivered by the reciprocating rakes will be dewatered to 79 or 84 pct solids.

Applications: It is safe to say that the hydroscillator is applicable to any problem requiring a clean rake product with the minimum amount of hydraulic water. It is a relatively new development in mechanical-hydraulic classifiers, and the range of applications, limited at present, is being expanded. The hydroscillator is well established in the closed-circuit grinding of ores and minerals and is a unit of relatively high capacity. This is evidenced by the fact that at Tennessee Copper Co. a 10-ft diam hydroscillator handled the entire mill tonnage of 2200 tons per 24 hr and overflowed this tonnage at 2 pct +48 mesh and approximately 54 pct −200 mesh. It is also interesting to note that the rake product contained only 1.0 to 2.0 pct −200, as compared to 9.0 to 10.0 pct for the conventional classifier. It is further significant that the average percent oversize in the grinding mill was 81 pct for the hydroscillator, in contrast to 68 pct for the conventional classifier circuit.

Although the hydroscillator is more expensive, it will find increasingly wide application not only for closed-circuit grinding but also for sharp two-product separations in beneficiation of iron ore and processing silica sand, to name but two of the prominent fields. A classifier that decreases the amount of undersize remaining in the rake product from the 130 tons left by the conventional classifier to 24 tons is bound to have many applications.

Centrifugal Classifiers

Limitations: 1—The solid bowl centrifuge. This unit can develop centrifugal forces up to 1000 times gravity, and with such characteristics it is the only commercial unit that will accomplish reasonably efficient separations down in the low micron range. Ordinarily the solid bowl centrifuge is limited to relatively fine separations from about 100 mesh to as low as 5 microns.

When the solid bowl centrifuge is used for dewatering, it cannot be expected to give a complete split of water and solids; the very fine material will be carried out with the overflow. In this connection it must also be kept in mind that as the removal of fines with the spiral product is increased the amount of moisture in this product will also increase, as it will in any of the mechanical-hydraulic classifiers. The feed volume must be kept to conservative proportions: the largest unit, 54x70 in., is limited to about 500 gpm.

2—The stationary cone or cyclone classifier. In this type of classifier the centrifugal force is set up by injection of the feed tangentially under pressure. Pressure is generally maintained at 15 to 20 psi but is raised up to as much as 80 to 100 psi when extremely fine separations are necessary.

The cyclone, having a steep-angle cone section of 20°, is in its present design usually limited to separations between 147 microns (100 mesh) on the coarse side and 10 to 20 microns on the fine side. It is limited in that abrasive action of any appreciable amount of coarse particles in the feed causes excessive wear when high pressures are used.

The stationary cone classifier, like the bowl centrifuge, will not have a clear overflow as long as slimes are present in the feed. A completely granular feed devoid of slimes would permit clear overflow, but such conditions are rare.

It is well to keep in mind that both the cyclone and the solid bowl centrifuge classify at heavy feed densities as compared to any of the other types employing an open pool for classification.

Larger diameter units favor the coarser separations, i.e., the 100 to 150 mesh range, whereas small diameter units are preferred for separations from 150 mesh to 10 microns.

The percent of solids in the feed has little effect on the underflow density, which will range from 55 to 70 pct, depending on the screen analysis fineness range. The finer the material, the lower the percent of solids.

An indication of capacity is a figure of 1000 to 1200 gpm for the 24-in. diam cone when it makes a 150-mesh separation on a material of 2.7 sp gr.

Applications: The stationary cone type of centrifugal classifier is applicable in a wide variety of fields. Undoubtedly it will be preferred in some cases that would require a large hydroseparator, since a combination of cones would eliminate the need for a large tank. There is good evidence of this in the phosphate rock washing operations in Florida.

As intimated above, the stationary centrifugal cone classifier is now accepted as a good primary deslimer. This simple centrifugal classifier has been launched in several other directions which appear to be promising: fine silica sand recovery, beneficiation of low-grade iron ores, and preparation of concentrator mill tailings for mine backfill.

The Cone Classifier

Limitations: An inverted cone with feed entering a centrally located feedwell at the top, the overflow spilling over the outer rim and the underflow discharging at the apex, makes a very simple classification device. Since no provision is made for scraping settled solids from the inside surface of the cone, a steep angle, 60° to 75°, must be used to minimize deposition. Hence the vertical clearance just about equals the diameter, and this may well become a disturbing factor when cones 8 to 12 ft in diam are called for. Probably the most common top size of cone is 8 ft in diam, but units of 10 to 12-ft diam have been installed.

The cone classifier, like the hydroseparator, provides only a quiescent pool for settling solids, and, as would be expected, effectiveness of classification is of low order, approaching closely that of hydroseparator. It is no secret that the operation of cone classifiers is sensitive to the character of feed solids. Any material containing fine particles having a tendency to adhere to a sloping side will build up on the sides of the cone, often plugging it entirely.

The cone works best when fed a solids mixture containing a high percentage of truly granular material which will scour the sides. With this kind of feed, dense underflows in the range of 40 to 60 pct solids can be maintained by any of a number of restricted apex discharge arrangements. The operation under dense underflow conditions is frequently critical. To prevent plugging the practice is to maintain free-flowing discharge at 35 pct solids.

There is practically no limitation on the size range of solids that can be handled in a cone classifier. Classification should not exceed 28 mesh on the coarse side and about 325 mesh on the fine side. Large feed volumes will require a multiplicity of units, thereby imposing some problems for equal feed distribution.

Applications: The cone classifier has one big advantage over the other types in that it is cheaper in first cost and comparatively cheap to install. It uses no power and maintenance is low. On materials of highly granular character it can serve as a primary classifier or dewaterer.

With the improvements in mechanical-hydraulic classifiers, there are no longer many fields in which cone classifiers are extensively used. In the production of concrete sand they are frequently used to sort and dewater small flows of specialty products, the cones being located over bins or storage piles. There are also a number of applications for cone classifiers in certain industrial processing operations handling relatively small volumes of feed flow.

The Sand Washer

Limitations: The sand washer, with its circular line of buckets dipping into the settling pool of a round tank, is a rough and ready unit that thrives on big tonnage. As a classifier it has narrow limitations separation-wise, owing to the pool area afforded by the design.

The sand washer should not be selected for separations except between the range of 28 mesh on the coarse end and 65 mesh on the fine end. It can be used on a feed mixture containing particles as coarse as 1 in. and by special arrangement up to 1½ to 2 in. Ordinarily the feed size range to a sand washer is ¼x0 in., which is typical of a concrete sand, but the buckets can be designed to handle the coarse sizes mentioned.

Capacity limitation for the 12-ft diam unit is about 125 tons per hr of bucket product when a 48-mesh separation is made on material of 2.65 sp gr. This capacity necessitates a feed at 2 to 1 dilution or 33 1/3 pct solids. In other words, the 12-ft sand washer is limited to about 1000 gpm overflow when making a 48-mesh separation.

The washed product as it is discharged from the buckets will contain 17 to 20 pct moisture (83 to 80 pct solids). This product can be handled on an inclined belt conveyor without creating a backwash down the belt.

Under the same conditions of feed and overflow the 7-ft and 9-ft diam sand washers are limited to 25 to 35 tons per hr and 45 to 65 tons respectively.

Applications: Sand washers, as the name implies, have been used almost exclusively for removal of silt and clay from banks and from natural sand deposits under water. The usual flowsheet involves screening the several sizes of gravel with the final —¼-in. sand plus all the water, silt, and clay being directed to the sand-washing and dewatering device. If the sand washer is used, some provision must be made, such as a settling box, to slough off sufficient water to maintain the feed to the sand washer at 2 to 1 dilution or 30 to 35 pct solids.

There is little promise of a wide range of application for the sand washer type of classifier. It is possible, however, that it could be used for high tonnage sand slime separations in mining operations, particularly in the disposal of tailings.

It can be seen that there is much overlapping of functions and that limitations cannot be clearly defined. More often than not information is relative and comparative. It should be emphasized again that classification is not an exact science.

Milling Kentucky Fluorspar Tailings

by LaMont West and Robert R. Walden

KENTUCKY'S first acid-grade fluorspar flotation mill, shown in Fig. 1, was placed in operation Aug. 1, 1952, by the Pennsylvania Salt Manufacturing Co. at Mexico, Ky. During 1951 a critical shortage of acid-grade fluorspar developed and it became necessary for the company to produce part of its fluorspar requirements.

In 1951 the company purchased the Crider and Lennen metallurgical fluorspar mill and an adjacent stockpile of tailing at Mexico, Ky. The tailing, assaying about 26 pct calcium fluoride, originated from log washer and jig mill concentrators in the area. As the mill did not have a fluorspar dryer or convenient rail facilities, the company leased the Burleson concentrator at Marion, Ky., where these facilities were available. At Marion, Ky., on the Illinois Central Railroad, dry acid-grade fluorspar is transported direct to the company's Calvert City, Ky., hydrofluoric acid plant.

L. WEST, Member AIME, is Superintendent, Pennsylvania Salt Manufacturing Co., Mexico, Ky. R. R. WALDEN is Senior Mechanical Engineer, Central Engineering Department, Pennsylvania Salt Manufacturing Co., Philadelphia.

Discussion on this paper, TP 3787B, may be sent (2 copies) to AIME before July 31, 1954. Manuscript, July 10, 1953. New York Meeting, February 1954.

The Mexico flotation plant was reconditioned and designed to produce acid-grade fluorspar from the jig tailing stockpile. The new plant flowsheet is shown in Fig. 2.

The ore feed, old tailing, which contains 10 pct of waste rock and coarse gravel assaying approximately 8 pct CaF_2, is bound together by sticky clay slimes and at first presented a problem in ore handling. A typical screen analysis and assay of the tailing is recorded in Table I. Study of the analysis

Table I. Screen Analysis of Kentucky Fluorspar Tailings

Screen Size, Mesh	Pct	CaF_2	SiO_2	$CaCo_3$	R_2O_3
On ⅛ in.	10.02	8.14	40.66	50.03	1.17
On 20	3.28	18.07	38.86	37.80	5.47
On 40	3.20	28.00	32.38	32.42	7.20
On 60	3.43	34.83	33.40	27.78	3.99
On 80	3.75	38.76	35.27	22.44	3.53
On 100	4.81	45.35	31.61	19.31	3.73
On 150	6.52	46.91	31.04	17.76	4.29
On 200	7.98	45.42	29.69	17.14	7.75
On 325	8.02	40.38	36.24	15.32	8.06
Through 325	48.99	18.12	53.32	13.67	14.89
Stockpile ore	100.0	26.04	43.32	21.62	9.02*

* The 9.02 pct R_2O_3 includes 0.40 pct sulphur.

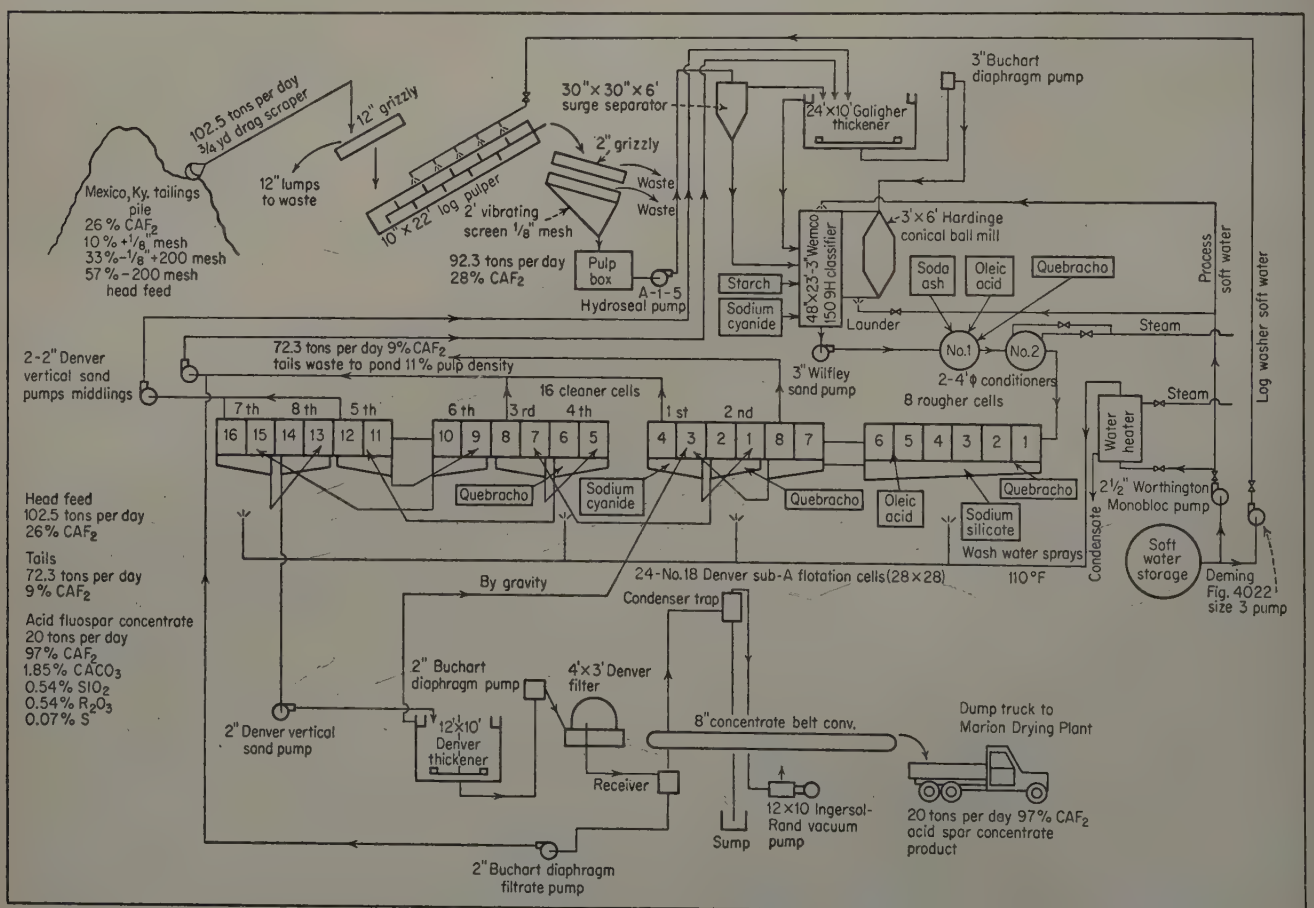


Fig. 1—A diagram of the acid-grade fluorspar flotation mill at Mexico, Ky.

suggested that the ore could be conveyed readily by pumping if it were slurried and screened to re-

Table II. Representative Screen Analysis of Classifier Overflow

Screen Size, Mesh	Pct
On 20	0.09
On 60	0.26
On 100	0.21
On 150	3.17
On 200	13.18
−200	83.08
Composite	100.00

move the +⅛ in. size, which is relatively barren of calcium fluoride.

Ore is pulped at 30 to 40 pct solids in a single log washer. Pulp overflows at the sand discharge-end through a 2-in. grizzly, then through a 2-ft Denver-Dillon vibrating screen with ⅛-in. mesh

Table III. Screen Analysis of Classifier Return Sands

Screen Size, Mesh	Pct
On 10	17.75
On 20	16.68
On 60	19.41
On 100	19.68
On 150	15.69
On 200	6.00
−200	4.79
Composite	100.00

wire cloth into a sump. The screen oversize is removed with a belt conveyor. A bulldozer daily removes the grizzly oversize and the belt discharge. A 3-in. hydroseal dredge-type pump moves the pulp to a surge cone. The surge cone discharges directly to a 48-in. No. 150 S.H. Wemco spiral classifier. The raw feed entering the classifier, which has been upgraded by screening off the oversize, assays about 28 pct CaF_2.

The density of the overflow from the classifier is maintained at from 15 to 17 pct solids. A characteristic screen analysis of the classifier overflow is given in Table II. Normally the circulating load of the grinding circuit is about 150 pct; Table III shows the screen analysis of the classifier return sands.

Ball-mill feed consists of classifier return sands and underflow from the flotation middling thickener. The middling thickener overflow constitutes part of the classifier makeup water. Analysis of the flotation middlings is given in Table IV.

The ball mill density, maintained at more than 60 pct solids, is controlled by the density of the feed from the middling underflow.

There are twenty-four No. 18 28x28-in. Denver Sub-A cells in the flotation circuit. The rougher consists of eight cells and there are eight stages of cleaners, consisting of two cells each. The cleaner cells are lined up in four groups of four cells each, arranged to effect two cleaning stages and to produce one middling to each group.

The plant produces approximately 20 tons of acid fluorspar concentrate daily, with recovery of approximately 76 pct. The analysis of the concentrate is recorded in Table V.

A 60-mesh stainless steel wire filter cloth on the concentrate filter resulted in an excessive loss of fines and an 80-mesh cloth was substituted to alleviate this condition. The original plant used a screw conveyor for wet filter cake handling; however,

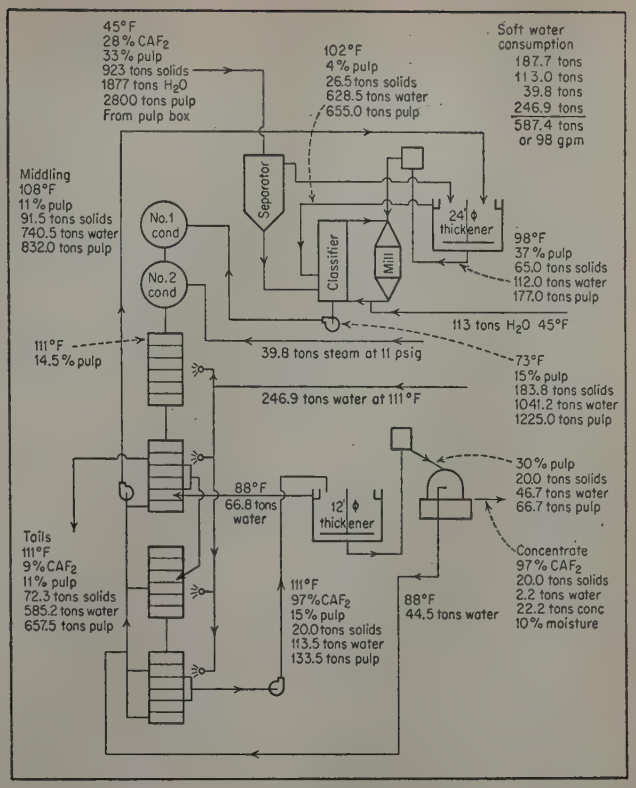


Fig. 2—The flowsheet for the reconditioned plant at Mexico, Ky.

maintenance was so great that it was replaced with a belt conveyor.

The acid spar concentrate after drying at the Marion, Ky., plant is bone dry. The 5-ft diam x 30-ft direct oil-fired rotary dryer was found to be very satisfactory when operating at temperatures slightly under 500°F. The dryer was completely lined with 4½-in. fire brick to cut down external heat radiation.

Table IV. Analysis of Flotation Middlings

Screen Size, Mesh	Pct	CaF_2, Pct	SiO_2, Pct	$CaCo_3$, Pct	R_2O_3, Pct	Fluorspar, Free or Attached, Pct
− 60 + 100 mesh	0.21	55.87	12.18	25.74	6.21	0.002
−100 + 150 mesh	2.16	60.74	5.07	31.81	2.38	0.040
−150 + 200 mesh	15.32	56.40	4.89	35.36	3.45	0.000
−200 + 325 mesh	51.56	52.90	8.28	32.80	6.02	0.000
−325	30.75	56.60	11.88	26.93	4.59	0.000
Composite	100.00	55.66	7.91	31.47	4.96	0.042

Water for the plant, obtained principally from local mines and from a drilled well, has a hardness of approximately 19 grains per gal. Recovery and

Table V. Analysis of Acid Fluorspar Concentrate

Item	Pct
Screen analysis	91.99 (−200 mesh)
Calcium fluoride	97.02
Calcium carbonate	1.85
Silica	0.54
R_2O_3	0.53
Sulphur	0.06

grade of concentrate were unsatisfactory and a 200-gpm Zeolite water softener was installed. All water now utilized in the process is dead soft as indicated by the soap test.

The pulp in the rougher cells is maintained at a temperature of 110° to 112°F and is controlled by a

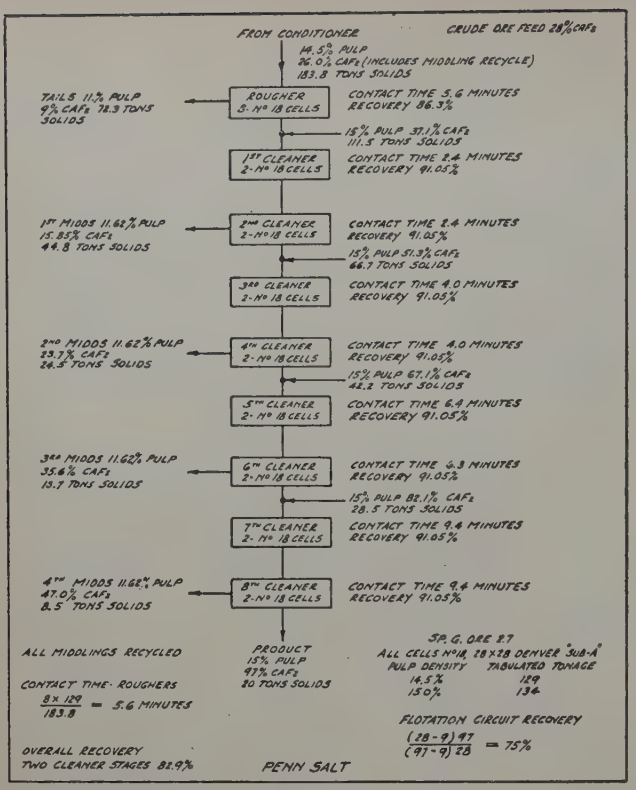


Fig. 3—Approximate concentrations, pulp densities, and contact time in the flotation cells as calculated. Note that the ratio of middlings is approximately 1 to 1.

direct injection of steam into the No. 2 conditioner. This temperature is also maintained throughout the flotation circuit by means of launder wash water at 110°F.

The classifier overflow being fed to the No. 1 conditioner is maintained at 15 to 17 pct solids, depending upon the quality of the stockpile ore being fed. The wide variation in the stockpile ore requires grinding changes (fineness) to liberate the calcium fluoride. This in turn requires a corresponding change in the classifier overflow and in the flotation cell densities.

A typical daily material balance is shown in Fig. 3. It should be noted that the ratio of middlings recycled to the feed is approximately 1 to 1. Fig. 4 shows the approximate concentrations, pulp densities, and contact time throughout the flotation cells. Data are calculated from actual operation.

Reagents

The flotation reagents used per ton of head feed are shown in Table VI. Inasmuch as the ore contains from 1 to 2 pct sulphides, sodium cyanide is fed into the classifier to depress the sulphides. The amount, approximately 0.4 lb of cyanide per ton of head feed, has been determined to be essentially correct for the ore being processed. If more cyanide than this amount is used, the pH increases, causing control of the pH by soda ash further on to become very critical, the flotation froth is toughened, and the sulphide content of concentrate is not further reduced.

Causticized starch is also fed to the classifier to depress silica. In preparing causticized starch to insure a colloidal suspension of the starch, a 1 to 1 ratio of starch to caustic is used. The starch is made up with a small amount of hot water and the caustic is added and stirred for several minutes before its dilution with hot or warm water. The caustic which is fed in association with the starch stabilizes flotation and renders the final pH control by the soda ash less critical.

Soda ash is fed into the No. 1 conditioner at a rate to maintain pH between 8 and 8.5. The No. 1 conditioner is also fed 0.3 lb of quebracho, 0.1 lb of sodium silicate, 0.5 lb of oleic acid, and a small amount of Pentasol No. 26 frother. These reagents are used to depress the calcite and silica and to act respectively as a flotation collector and frother for the calcium fluoride.

Sodium silicate and quebracho are also fed to the rougher concentrate at the rates of 0.1 and 0.2 lb, and 0.1 lb of oleic acid is fed as required into the overflow between the No. 5 and No. 6 rougher cells. Quebracho is fed as needed, 0.1 to 0.2 lb, at two points equally distributed along the flotation cleaning circuits. Provision was made to feed sodium cyanide into the launder of the first cleaner stage as may be found necessary.

Regardless of how well the ore is mixed on the stockpile with a bulldozer, there are wide variations in the flotation characteristics of the ore. The amount of mine plant refuse, alluvial soil, and organic material also varies considerably, rendering the ore most difficult to treat. For these reasons, it has not been possible to set operating conditions for more than a week at a time. Classifier densities and the quantities of depressants must be altered to meet the changing conditions.

Table VI. Flotation Reagents Per Ton of Head Feed

Reagents	Lb Per Ton
Oleic acid	8 to 14
Quebracho	0.4 to 0.6
Starch	0.7
Starch	0.5
Sodium silicate	0.4 to 0.5
Sodium cyanide	0.3 to 0.4
Caustic	0.5

When either the silica or the calcium carbonate become too difficult to hold within the concentrate specifications, the blend from the stockpile is changed.

Operation

During the initial weeks of operation, the total head feed was thickened and fed directly to the ball mill to repolish the mineral surfaces. This was believed necessary because the feed consisted of tailings that had been exposed to weathering conditions. The procedure actually increased metallurgical difficulty. Capacity was low and recovery below 65 pct, and it was difficult to maintain concentrate grade.

When the process was changed to that of directly feeding the classifier, with only the coarse sands going to the ball mill together with the recycled middlings, the metallurgy smoothed out and capacity was increased.

A more recent curative measure has been to cut out the No. 1 middlings and impound for remilling at a later date. This procedure enables the circuit to be bled of silica, which has a tendency to build up in the system.

Acknowledgment

The authors have endeavored to pass on experience gained in the design and operation of an acid-grade fluorspar flotation plant operating on log washer sands and jig mill tailings. They acknowledge gratefully the assistance of the plant personnel during preparation of this article.

Drilling Practice In Swedish Mining

by Ingvar Janelid

Drill jumbo at Kiruna uses two chain-fed drifters for drilling fanholes in top slicing. The drills are arranged to turn around a horizontal shaft and can be placed at any angle in relation to the shaft so the drillhole will have the desired direction.

DURING the last ten years, in the effort to save manpower and costs, methods of drilling and blasting in Sweden have changed and developed in a revolutionary manner. These developments have been accompanied by extensive alterations in methods of tunneling and in mining technique.

Since the end of the 1940's there has been a 100 pct change-over in Sweden to the use of tungsten carbide tipped drill steel. Both investigations and practical results have shown clearly that a tungsten carbide chisel bit fastened directly on the drill steel is the most economical for Swedish conditions. This applies to drill steel up to the longest size it is practical to handle. In terms of drilling footage, durability of the drill steel and the tungsten carbide bit is generally the same. In this respect, therefore, a detachable bit offers no advantage over integral steel. Transportation of integral steel to the grinding plant, instead of detachable bits only, involves heavier work, but this is counteracted by placing grinding plants closer together or by grinding at the working place itself. The drilling rate with integral steel is higher, the trouble of changing bits is avoided, and the difficulties which always arise with joints are eliminated.

At present steel dimensions are usually 7/8 in. with a shank 4¼ in. long, whereas the international standard shank was formerly 3¼ in. The longer shank renders possible a better guide control of the steel and consequently longer life for the rotation chuck and the water-flushing pipe. After extensive experiments and investigations, agreement has been reached in Sweden on certain series of standards, shown in Table I. Other steel dimensions are, of course, available for special purposes. The standard drill-steel set begins with a 34-mm diam and has a length interval of 2 2/3 ft. On the other hand, for ordinary drifting it has been proved more economical to use greater length intervals on the steel, so only one steel change is required for a 6½-ft round. Moreover, owing to the relatively close hole spacing necessary in a drift round, it has been possible to reduce the diameters of the tungsten carbide bits. This results in faster and cheaper drilling. As shown by the table, the lengths of drill steels for normal drifting are 3¾ ft and 7½ ft, with 29 and 28-mm diam bits.

During the last few years operators have begun to use ¾-in. drill steel in place of the 7/8-in. drill steel chiefly employed earlier. This is a valuable development in drilling technique, particularly in view of

I. JANELID, Member AIME, is Professor of Mining and Surveying, Royal Institute of Technology, Stockholm, Sweden.

Discussion on this paper, TP 3801A, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Feb. 1, 1954. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 544. The intervening non-Transactions pages appeared in Mining Engineering.

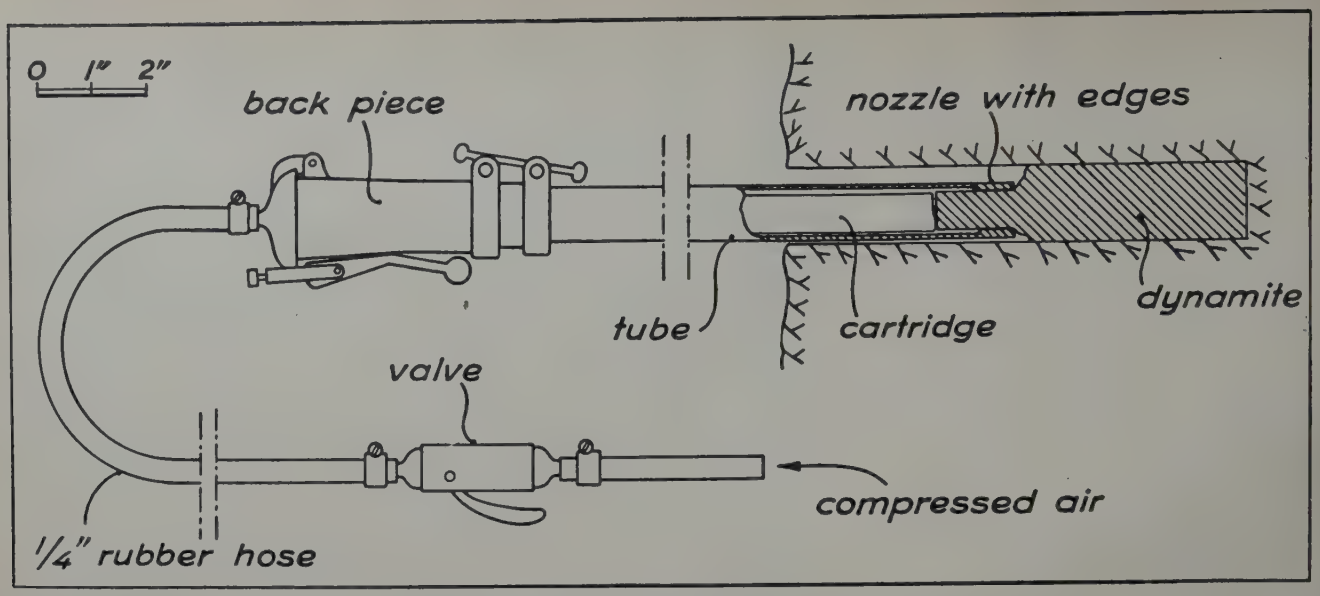


Fig. 1—Diagram of pneumatic apparatus for loading boreholes.

Table I. Standard Tungsten Carbide Tipped Steel Used in Sweden

Integral Steel						Flexible Steel	
7/8 In. Hexagon							
Normal Series		For Drifting		3/4-In. Hexagon		½ x 1 In.	
Length	Bit Diam, mm	Length	Bit Diam, mm	Length	Bit Diam, mm	Length	Bit Diam, mm
2 ft 7 in.	34	3 ft 8 in.	29	1 ft 4 in.	29	2 ft 7 in.	34
5 ft 3 in.	33	7 ft 4 in.	28	2 ft 7 in.	28	5 ft 3 in.	33
7 ft 10 in.	32			5 ft 3 in.	27	7 ft 10 in.	32.5
10 ft 6 in.	31			7 ft 10 in.	26	10 ft 6 in.	32
13 ft 1 in.	30			10 ft 6 in.	25	13 ft 1 in.	31.5
15 ft 9 in.	29			13 ft 1 in.	24	15 ft 9 in.	31
						18 ft 4 in.	30.5
						21 ft	30
						23 ft 7 in.	29.5
						26 ft 3 in.	29
						28 ft 10 in.	28.5
						31 ft 6 in.	28

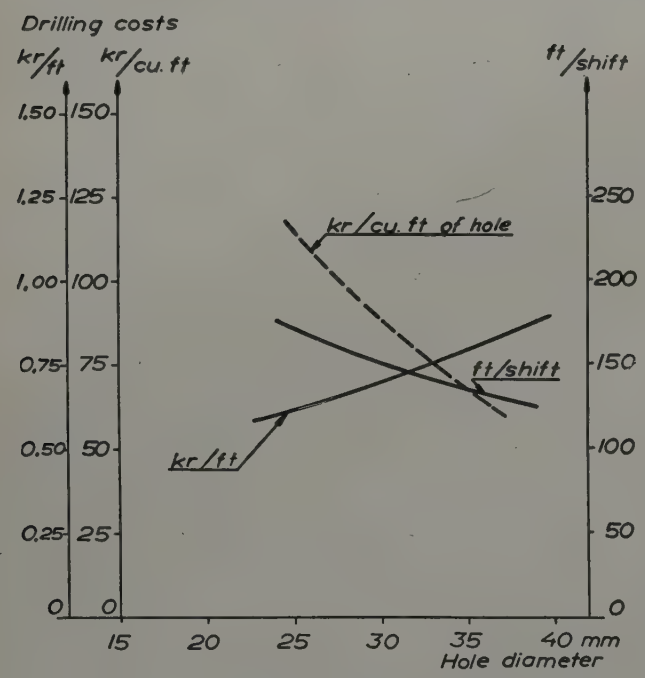


Fig. 2—Effect and costs of drilling in hard iron ore.

the fact that larger drill steels are used in countries other than Sweden, especially in America. Steel dimensions in Sweden at the present time are of the following proportions: 2 pct 1 in., 85 pct 7/8 in., and 13 pct 3/4 in., with steady increase in use of the smallest dimensions.

With 3/4-in. steel smaller bit diameters are used, beginning with 28 mm (see Table I), which of course means a reduced charging volume, reckoned per drilled foot. In spite of this, the smaller sizes have proved in many cases to be economically preferable in hard and abrasive rock. When an investigation is made, for instance, of how the charging volume is actually used in a 36-mm drillhole that can hold about 0.94 lb dynamite per ft, it is discovered that a normal charge comprising 25-mm cartridges, loaded with a stick, gives about 0.34 lb of dynamite per ft of drillhole. In other words, only about 35 pct of the drillhole volume is utilized, which undoubtedly will surprise many people. If charging is done more carefully the hole spacing can be retained and a smaller drillhole diameter adopted. To obtain a satisfactory charging density two different methods have been employed: 1—charging with 5-ft long cartridges, which have a larger diameter and fit the drill hole better, and 2—charging by means of a special pneumatic apparatus manufactured by the Nitroglycerin AB in Stockholm. The following is a practical example of the charge per foot obtained in 36-mm boreholes at the Malmberget mines:

Loading normal cartridges with wooden stick	0.34 lb dynamite per ft drill hole
Loading 5-ft long cartridges of larger diameter	0.58 lb dynamite per ft drill hole
Loading by means of pneumatic apparatus	0.81 lb dynamite per ft drill hole

The pneumatic charging apparatus is shown in Fig. 1. The manner in which it operates is briefly described here.

The charging pipe is equipped with a back piece which can be opened and closed and through which the cartridges of explosives can be inserted. The diameter of the cartridges must be 1 to 2 mm smaller than the internal diameter of the pipe so a current of air can pass between the cartridges and the wall of the pipe. Compressed air is supplied through a hose connected to the back piece. A choke flange in the air-intake limits the amount of air admitted so the cartridge will not exceed a maximum speed of 33 ft per sec.

The pipe is equipped with a nozzle in the front end, the internal diameter of which is the same as the diameter of the cartridge. When the compressed air is turned on, the current of air draws the cartridges with it until the front one reaches the nozzle. The air is thereby hindered from streaming out. The pressure in the pipe rises and the cartridge is forced out into the borehole, whereupon three steel edges on the inside of the nozzle cut up the paper casing of the cartridge with lengthwise cuts. The slit cartridge can easily be packed together with the help of the charging pipe so that the dynamite fills the hole completely. The pipe is then drawn out and the next cartridge is pressed forward and packed in the same manner.

The durability of ¾-in. steel is somewhat inferior to that of ⅞-in. steel, but the wear on the tungsten carbide bit per drilled foot is considerably less with the smaller bit diameter. Use of 28-mm bits instead of 34-mm bits almost doubles drilling footage per grinding in hard rock.

In the diagram, Fig. 2, the total drilling costs, together with the drilling footage per shift, are plotted as a function of the drillhole diameter when hard ore is drilled. As shown, decreasing the drillhole diameter increases the total drilling footage per shift and decreases cost per drilled foot, but the cost per volume unit of drillhole increases. If the same amount of explosives can be packed in a smaller drillhole by improved charging methods, and the hole spacing can be retained, the total cost will be lower.

One of the most important factors in choosing the hole diameter is the frequency of boulders, which influences the costs for secondary blasting, loading, transportation, and primary crushing. The boulder frequency can be effectively controlled by close hole spacing. The advantage of using ¾-in. steel and small hole diameters is fast and inexpensive drilling. However, charging must be done carefully, preferably with the loading apparatus.

A summary of the points that are of chief importance in the choice of a suitable drillhole diameter can be expressed as follows. A large borehole gives

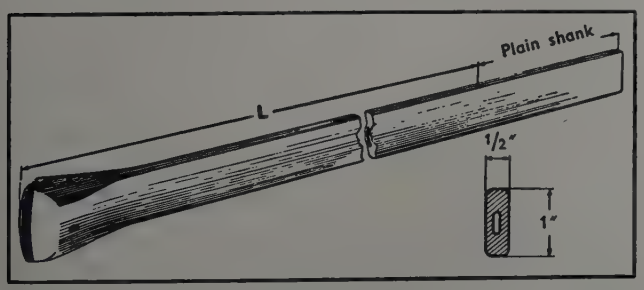


Fig. 3—Flexible drill steel.

the cheapest borehole volume, but when consideration is given to boulder frequency and possible hole spacing, for instance, in tunnels or in narrow orebodies, the hole spacing must be comparatively close. Hole diameters must not be larger than necessary for charging the calculated amount of explosives.

Use of ¾-in. steel, especially in hard rock, is steadily increasing. An examination of its practical and economic advantages in underground mining has been made by G. Nilsson, the chief mining engineer at Striberg, and by others. Among the mines which have gone over partly to ¾-in. steel are the Stripa, Garpenberg, Haksberg, and Boliden.

As previously mentioned, detachable bits have not been used in Sweden other than for long holes. A

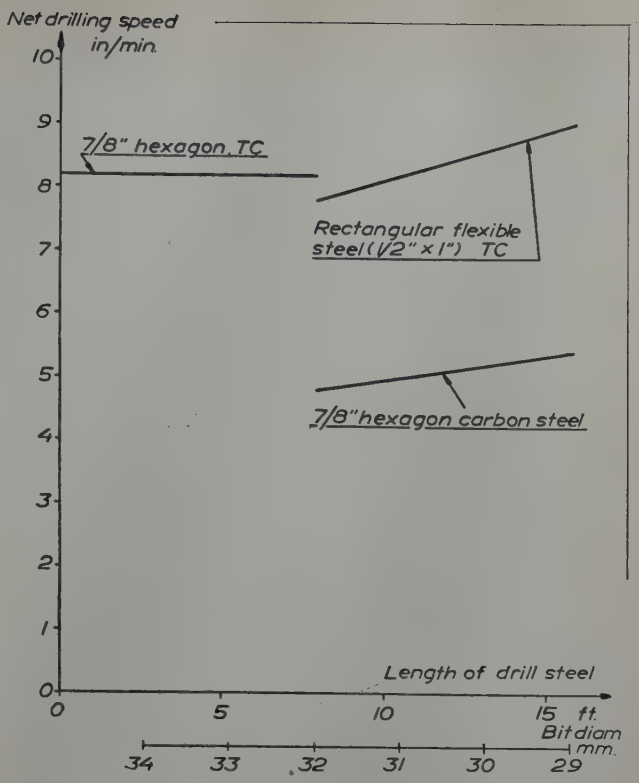


Fig. 4—Net drilling speed in iron ore with different types of drill steel. Rock drill: 2 9/16 in. with pusher.

completely new type of drill steel has been introduced for holes of 13 to 26 ft long, namely flexible steel, which can be bent both during transportation and when it is inserted in the hole. Dimensions of this steel are ½ to 1 in., see Fig. 3. The drilling rate with the same diameters of tungsten carbide bits is somewhat lower with flexible steel than it is with ⅞-in. steel.

A fair comparison of the respective drilling rates of flexible steel and extension steel is impracticable, as flexible steel requires small light machines and extension steel heavier machines with power feed.

At Grängesberg, where more than a million tons of iron ore are mined per year with top slicing, it is very important to be able to drill some of the holes longer than is possible with ordinary integral steel. At the same time, of course, it is desirable to retain the same light drilling outfits normally used. Development costs will be less because the height between slices and distances between crosscuts may be greater than if only ⅞-in. integral steel is used. Formerly, bendable carbon steel was used in Grängesberg for the longest holes, but flexible steel has now been adopted there for holes 8 to 20 ft. Fig. 4 shows the footage obtained with ⅞-in. hexagonal steel and with ½x1-in. flexible steel, both with tungsten carbide bits. The diagram also shows the footage obtained with ordinary bendable ⅞-in. carbon steel, which is not suitable for tungsten carbide bits. As is shown in the diagram, the drilling rate is very good for flexible steel and higher than for ⅞-in. steel with tungsten carbide bits, because of the smaller bit diameter.

Fig. 5 shows how flexible drill steel can be bent when inserted in a drill hole. In the near future flexible steel will probably be used widely as a complement to normal steel.

Long-hole drilling is now a customary method, especially in America; nevertheless, the following information may be of interest. Both diamond drill-

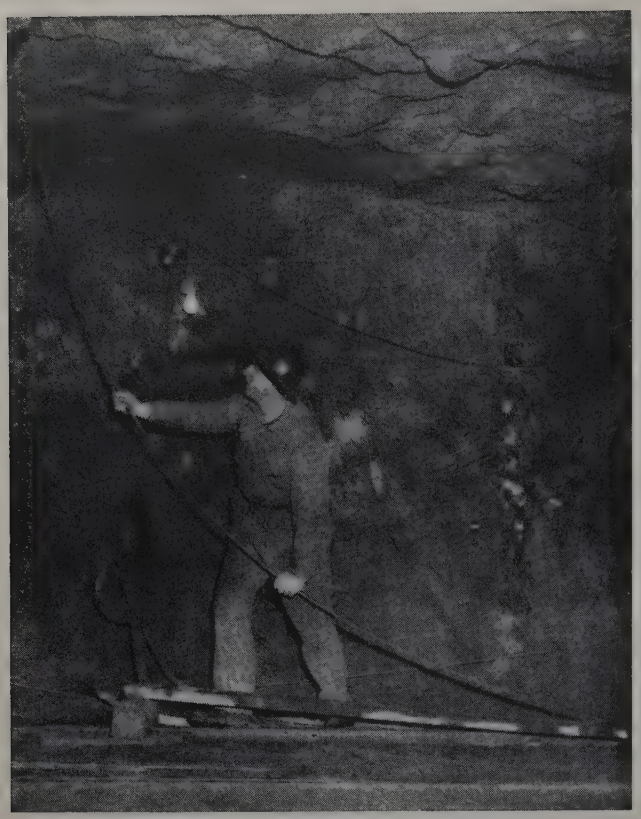


Fig. 5—The use of flexible steel in top slicing.

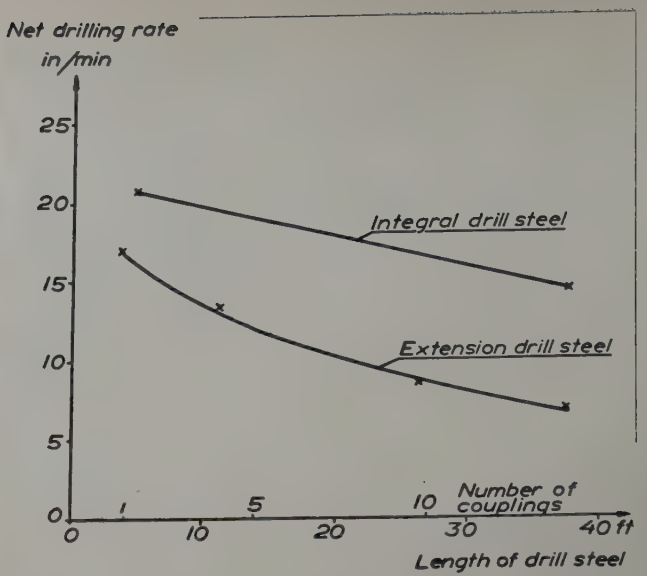


Fig. 6—Net drilling rate as a function of length of drill steel. A 4-in. drifter with chain-feed and a bit 40 mm in diam are used.

ing and extension steel (see Fig. 6) with tungsten carbide detachable bits are used for long-hole drilling in Sweden.

The diamond drill machines are driven by compressed air and are of two different types, namely, those with mechanical force feeding and those with automatic friction feeding. For drilling in hard rock the latter type has proved to be preferable as it causes less wear on the drill bits. In loose and medium hard rock noncoring bits with small diamonds are used, but in hard rock core bits are the most economical and considerably reduce diamond wear. Total footage in core drilling is decreased by reason of the necessary core removal, as well as interruptions when the core breaks. Considerably better results have been obtained through reversed water flushing, whereby the core as well as loose core fragments are pressed back into the drill rod. The flush water is forced through a special sleeve on the outside of the drill rod, or a special double-tube core barrel is used. In certain cases the drill rod is given such a large diameter that the core can be flushed back through the entire rod, and in this case core removal takes place less frequently. To facilitate the flushing out of the core compressed air is occasionally added to the flush water. The reason for this is that when the air expands while passing out of the drill rod the water draws the core as well as the core fragments along more readily. In this connection it has proved advantageous, when using hole diameters of 36 mm, to reduce the core diameter from 22 to 18 mm. Another very great advantage with reversed water flushing is reduced diamond wear, sometimes only half in comparison with customary flushing; this is due to the fact that no core fragments are pressed forward to the diamond bit.

During the last few years long-hole drilling with *pneumatic hammer* drills and extension steel has come more and more into the foreground and partly replaced diamond drilling. Drill rods, which are usually ⅞ in., have become more durable owing to the use of longer couplings, among other reasons. The threads have also been designed in a form with higher pitch, making it possible for uncoupling to be done conveniently by hand without any tools. Larger and stronger machines, such as the Atlas Diesel 4-in. BBC 41 RBL, see Fig. 7, are used for long-hole drilling. Power feeding with a compressed air motor is adopted.

When holes are drilled with extension steels a part of the energy transmitted is lost in the joints; consequently the drilling footage is lower than with integral steel. In the diagram, Fig. 6, a comparison has been made between drilling with integral steel and extension steel with the same machine. Note that a higher output is obtained with integral steel. This fact is taken advantage of to the fullest extent at working places where long drill steel can be used, as in tunnels, large spaces underground, and, of course, open-pit stoping.

In Malmberget, where a large part of the ore is mined by long-hole drilling, excellent material has been obtained for comparison between diamond drilling and drilling with extension steel. Drillholes had an average depth of 100 ft. Drilling footage per shift in ore is about 50 pct greater with pneumatic drills and extension steel, and drilling costs are 20 to 30

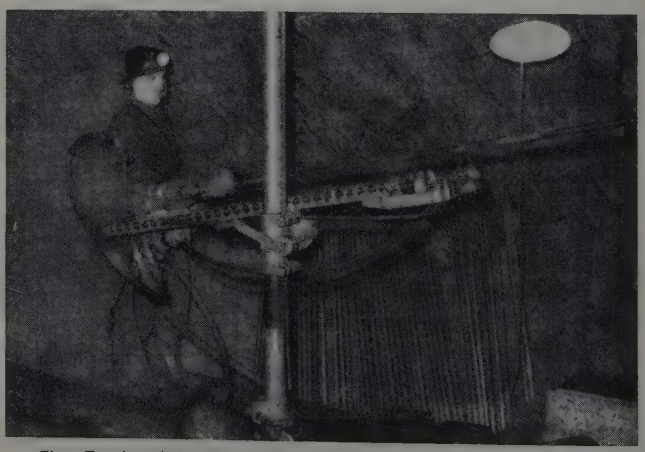


Fig. 7—An Atlas Diesel 4-in. drifter with motor feed for long-hole drilling.

pct lower than with diamond machines. With the present equipment for long-hole drilling in medium hard rock, it will be cheapest to use pneumatic drills and extension steel up to 130 or 160 ft.

Before the different types of rock drills are described attention should be called to the fact that there are drilling sites requiring relatively few holes and others requiring mass production of drillholes. In the first case the practice of one man to one machine has been accepted throughout Sweden; the simplest proof of this is that since 1948 no new stopehammers have been manufactured in Sweden, but only light rotary machines and pushers. The endeavor has been to make a simple and inexpensive machine which can be used under many different conditions. The method of using light machines and pushers is too well known in the U. S. to need further description here.

In the second case, i.e., with mass production of drillholes more or less at the same working site, power-fed drilling machines mounted on wheels or easily transported are usually preferable. Through the introduction of short delay blasting of large rounds in such operations as cut-and-fill and shrinkage stoping, these power-fed machines have come into more general use during recent years. Fig. 8, a picture of cut-and-fill mining in Boliden, shows a wagon drill with two chain-fed drilling machines, placed side by side, which are operated by one man. The footage has been increased from 180 ft with a pusher to 550 ft per manshift with the twin machines. By the use of more powerful feeding, the drilling rate has been increased from 12 to 16½ in. per min. It should be pointed out, however, that the pusher drill is not very suitable for overhead drilling.

In drifting comparative investigations have been made between a drillmobile equipped with three chain-fed drifters and a self-propelled platform in combination with three pusher drills, see Fig. 9. The drilling platform, which runs on rubber wheels and is driven by a compressed air motor, is very easily moved. On completion of drilling with the platform in the position shown in Fig. 9, the platform is moved to the opposite side of the drift. Results shown in Table II were obtained in Kiruna from a test in a gallery 10x16½ ft.

Although the drillmobile is easily moved and easily operated, equal or better results have been obtained hitherto with the cheaper pusher drills in combination with a simple drilling platform.

For drilling fanholes in a top slicing a special drill jumbo has been developed. See cut, p. 613, showing top slicing in Kiruna. Two chain-fed drills, arranged to turn around a horizontal shaft, can be

Fig. 8—An illustration of cut-and-fill mining at Boliden. The wagon drill with two chain-fed drifters, shown above, is operated by one man. With these twin machines footage has been increased from 180 ft with a pusher to 550 ft per manshift.

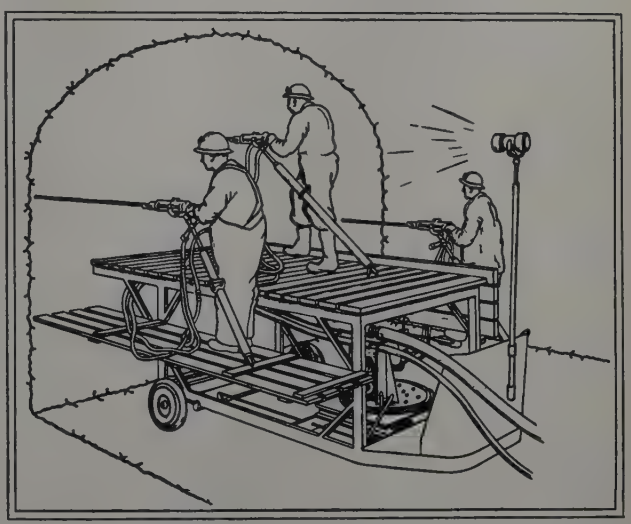
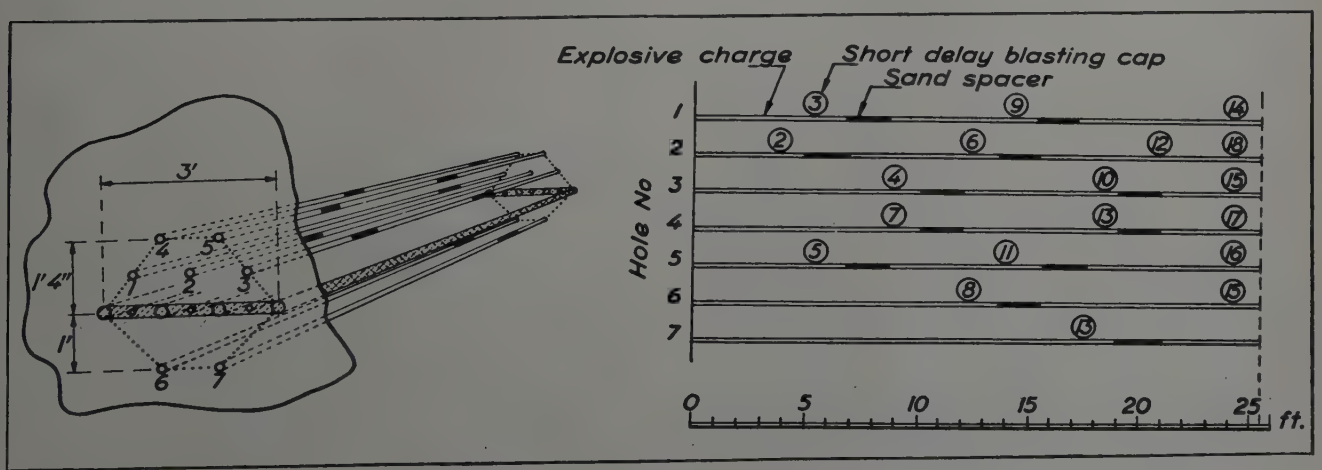


Fig. 9 (above)—Drilling platform for pneumatic pusher rock drills. The drift is 16 ft 5 in. wide and 11 ft 5 in. high.

Fig. 10 (below)—The multi-burn cut and a diagram of charges in drillholes.



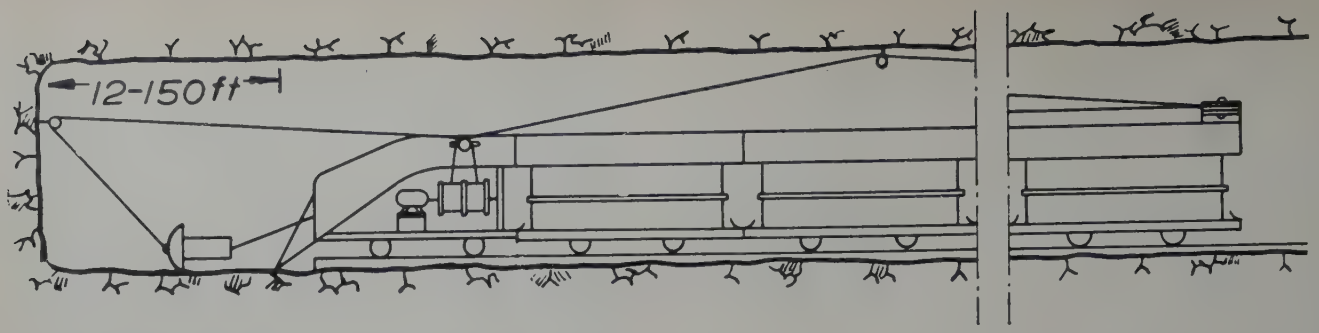


Fig. 11—Scraper equipment for small tunnels, with cars for an entire round. The scraper is drawn along flanges on top of the cars. The scraper ramp is shifted every 40 or 50 ft of advance.

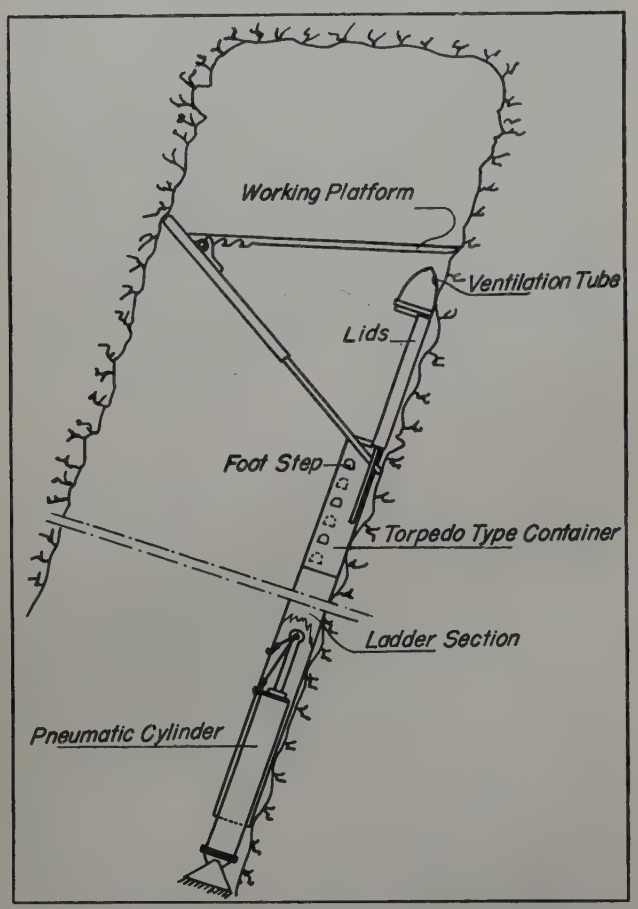


Fig. 12—Use of a steel ladder for driving raises.

placed at any given angle in relation to the shaft so the drillhole will have the desired direction.

All investigations and new ideas for raising the efficiency of drilling and blasting operations must be based on a thorough study of the most favorable utilization of personnel and machines. Results reported by different companies, summarized in this paper, have been obtained after very comprehensive time studies. The effort to render rock blasting more efficient is expedited when progress and results obtained at one place are made available to others. For this reason a collection of articles on problems of blasting is published in Sweden under the title *Manual on Rock Blasting*,[1] which is printed in four languages.

Table II. Results of Test Drilling in a 10x16½-Ft Gallery at Kiruna

Item	Drilling Platform with Three Pusher Drills	Drill-mobile with Three Hydraulically Controlled Machines
Drift, ft	840	700
Stope, cu ft	6113	742
Total working time, hr	2020	2181
Manhours for drilling per foot of advance	2.5	3.3

In every field, it is true, the cheapest products are obtained by mass production, where the setting-up time between the various working steps is the shortest possible per unit produced. In rock blasting, which is a combination of completely different forms of work, an attempt is made to achieve high efficiency of drilling and blasting as well as of loading. However, the work must be so arranged that the working cycle is as long as possible. This can be brought about partly by drilling long holes and partly by drilling a large number of holes for each round.

Practical Examples

In cut-and-fill mining in Boliden, Fig. 8, it has been possible to increase the mining results considerably by drilling very large rounds. It is estimated that in shrinkage stoping in Kiruna it may be possible to increase capacity as much as ten times by blasting large rounds rather than by using the old method of blasting. Long-hole drilling at Malmberget has been described in an earlier paper.[2]

To obtain a long working cycle in drifts and tunnels the advance per round must be increased. However, the advance per round depends wholly on the length of the cut and therefore much experimenting has been undertaken to solve this problem. Two different cuts have been tried: 1—the burn cut or Michigan cut with large diameter center holes, and 2—a special type of burn cut with divided charges in every hole, together with short-delay blasting.

Originally the method employing short-delay blasting was done with the charges divided into only two parts per hole. A narrow cut of about 13 ft deep could be produced. Later on Langefors of the Nitroglycerin AB developed a system for dividing the charges, whereby cuts up to 23 ft deep could be obtained. The principle of this method, which is known as the *multi-burn cut*, is presented in Fig. 10. The four large drill holes in the center (diam 80 mm) are not charged, and the three smaller holes (34 mm) between the larger ones have column charges and are fired first to produce an opening in the middle of the cut. In the other drill holes, Nos. 1 to 7, the charges are divided, according to a predetermined system, by sand spacers of about 2 ft long. Blasting the divided charges with short-delay caps placed in a predetermined sequence breaks out the rock a little at a time. The method has recently been used very successfully in blasting out underground air raid shelters and oil tanks.

When the Michigan cut with one or more large center holes is used it has been found that the large

Fig. 13—Working platform and torpedo containers with lids open, seen from underneath.

holes must be of 4 to 6 in. diam if good results are to be obtained. Pneumatic hammers are used to drill the large holes directly or to ream out normal smaller holes. Several different types of drill bits have been tried for this purpose.

A necessary condition for using the above-mentioned burn cuts with parallel and closely placed holes is that the drill holes must be correctly directed from the beginning and drilled straight. At the present time the possibilities for this seem to be satisfactory.

In all drifting it is essential to a rapid advance that the work be organized so that all setting-up times and delays are as short as possible. Boliden Co. has constructed a type of scraper equipment, shown in Fig. 11, which makes quick loading and transportation possible in drifts with a cross-section of about 50 sq ft or more. The wagons are so constructed that the scraper slides along their upper edges and fills the whole train, beginning with the wagon which stands nearest the scraper. The scraper ramp is placed first, 13 to 20 ft from the drift face, and is allowed to remain there until the scraper length is about 160 ft. All drilling is done with pusher drills, which are easily moved away to a protected place behind the scraper hoist during blasting. The advance with this outfit has amounted to 23 ft per day of two shifts, five men to the shift, in a tunnel area of 97 sq ft. Two men handle the scraper. The rock is first removed from the face to enable the other three men to start drilling. Very thorough comparative investigations in connection with this and other methods have been undertaken by V. Wanhainen of the State Power Board in Sweden. Results will be published in the *Manual on Rock Blasting.*

Another novelty which may be mentioned here is a steel ladder, Fig. 12, used in driving raises. The ladder can best be employed in inclined raises deviating at least 20° from the vertical. The lower part of the steel ladder consists of a powerful air cylinder, which can push up the whole construction so that a new section can be added. A working platform is arranged over the top of the ladder. The upper section of the steel ladder consists of a strong torpedo-shaped cylinder with lids. The platform planks and drilling equipment can be kept in this cylinder during blasting. The semi-cylindrical sheet metal tube serves as a ladder owing to the provision of notches formed in the metal. Air and water hoses and electric cables run inside the tube. The entire construction is so strongly dimensioned that it will not be seriously damaged by falling rocks.

In a comparison of the new method of driving raises with the older, more conventional method using timbering, it is found that:

1—The setting-up time for drilling a round decreases from 2½ to 1¼ hr.

2—The cost of material for a ladder 130 ft high is 4500 Swedish kronon. The ladder can be used for at least five raises. This means a cost of 900 Swedish kronon per raise, in comparison with 3500 Swedish kronon per raise for timbering.

3—The saving of time in raising is considerable, since both the work of timbering and removing the timber afterwards is eliminated. The pipes are simply led through the steel ladder, and ventilation takes place quickly because fresh air can be brought near the top of the raise.

4—Safety is increased when this steel ladder is used.

Summary

In hard rock ¾-in. drill steel and small diameter bits have been shown to be more economical than normal ⅞-in. steel. Flexible steel is used to great advantage instead of extension steel when holes between 13 and 26 ft are drilled. It is more economical to use extension steel than diamond drills in drilling holes up to 100 or 130 ft in medium-hard rock. Small pusher drills are the usual type, but in long-hole drilling and in mass production of holes in a limited area the larger 4-in. machines with chain feed are used. By means of special blasting methods, great progress in tunneling is being made with long advances per round. A mechanical steel ladder has been developed for raising which reduces the required working time, increases the production per shift, and insures greater safety.

References

[1] K. H. Fraenkel (Chief editor): *Manual on Rock Blasting.* AB Atlas Diesel, Stockholm, and Sandvikens Jernverk AB, Sandviken, 1952.

[2] B. Hjortzberg-Nordlund: Underground Mining Trends in the Great Swedish Export Iron Mines. *Trans.* AIME (February 1952) **193**, pp. 187-192.

[3] C. A. Widén and W. Haglund: Rock Drilling with Tungsten Carbide Bits. *Teknisk Tidskrift* (1945) p. 533.

[4] Erik Ryd: Tungsten Carbide Bits for Rock Drilling. *Jernkontorets Annaler* (1947) No. 9, p. 373.

[5] C. O. Didring and S. Åberg: Drilling with Steel and Tungsten Carbide Bits with Different Types of Rock Drills. *Teknisk Tidskrift* (1947) p. 359.

[6] C. G. Carlström: Advance per Round in Drifting with Tungsten Carbide Bits. *Teknisk Tidskrift* (1948) p. 821.

[7] T. Ekstam, K. H. Fraenkel and E. Ryd: Rock Drilling with Tungsten Carbide Bits. *Jernkontorets Annaler* (1949) No. 8, p. 253.

[8] Gunnar Ryhre and Åke Kallin: Rock Drilling with Tungsten Carbide, and Its Economy. *Teknisk Tidskrift* (1949) p. 553.

[9] I. Janelid: Open-Pit Stoping. *Teknisk Tidskrift* (1951) No. 4.

[10] I. Janelid: Swedish Developments in Drilling and Blasting. Lecture at NIM 4, given in Helsingfors, June 1951.

[11] T. Ekstam and E. Ryd: Rock Drilling with Tungsten Carbide Bits. *Jernkontorets Annaler* (1952) **136.**

The Liquid-Solid Cyclone as a Classifier In the Closed-Circuit Grinding of Concentrates

by F. M. Lewis and E. C. Johnson

Used as a classifier in a plant expanding capacity or changing to closed-circuit operation, the liquid-solid cyclone offers the advantage of being adaptable to existing conditions. This paper presents the costs involved and experience gained from operating a unit of this kind.

BY now application of the liquid-solid cyclone to various classification problems in the chemical and metallurgical fields is more or less familiar. A less publicized application receiving wider consideration as time goes on is its use as a classifier in closed-circuit grinding operations. This paper presents the costs involved and the experience gained from operating a unit of this kind.

In May 1951 the Tennessee Copper Co. installed in its London mill at Copperhill, Tenn., a liquid-solid cyclone in closed circuit with a 6x12-ft ball mill. The circuit in which this cyclone is used is the regrind or secondary grinding circuit of a copper flotation plant. Originally it consisted of a 7x1-ft conical ball mill in closed circuit with a bowl classifier. Changes made in the primary grinding circuit of the mill necessitated a regrind circuit of greater capacity. The bulk concentrate to be ground was raised from 900 or 1000 tpd to 1200 or 1300 tpd, and to accommodate this increase a 6x12-ft ball mill which had been in the primary grinding circuit was moved into the regrind circuit. When this was done it was found that the classifier was too small to give efficient operation. Space in the plant was at a premium, and since use of the cyclone offered considerable economy the installation shown in Figs. 1-3 was made.

The unit installed is a 24-in. diam cyclone classifier with a 20° cone angle. Diameter of the vortex finder is 8 in., the feed entrance area 20 sq. in. Maximum diameter of the adjustable apex valve is 6 in. and minimum diameter approximately 3.5 in. To refer again to Figs. 1 and 2, the cyclone* is mounted horizontally, and the underflow, or sands product, discharges directly into a 6x12 ball mill. This mill has a speed of 21.9 rpm and is powered by a 200-hp motor. It is charged with 18 tons of 1-in. balls. The overflow, or fines, passes by gravity directly to the copper flotation plant. The ball mill discharge and the new feed combine in a sump from which they are sent to the cone by an 8x6 SRL pump at a rate of 497 gpm. Pressure at the cyclone is 5 psi.

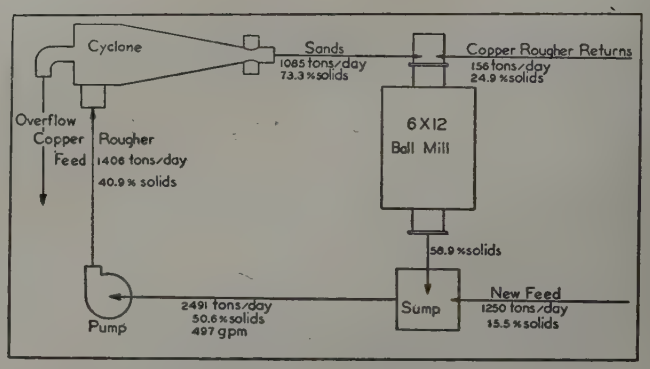


Fig. 1—Closed-circuit grinding of copper flotation feed using 24-in. cyclone.

For reasons dictated by the nature of the ore treated, particularly the bonding of fine grains of chalcopyrite to pyrrhotite, the froth from the second cell of the copper rougher circuit is returned to the regrind mill. This flow, which somewhat complicates the standard closed-circuit calculation, is seen in Fig. 1 to be 156 tpd of solids. This combines with the cyclone classifier underflow to constitute the ball mill feed.

The requirement of the regrind product is that it be 77 to 80 pct —200 mesh. The liquid-solids cyclone operating in conjunction with the 6x12-ft mill adequately met this requirement. Of primary importance is the fact that this arrangement provided sufficient classification capacity to replace the undersized classifier and was capable of building up a circulating load to 87 pct, large enough to give an efficient grind. In this instance the circulating load is taken as the ratio of sands to new feed.

Any comparison of performance between this liquid-solid cyclone and the replaced classifier would of course be unfair, since the bowl classifier was

* The cyclone discussed in this paper is the Dorrclone, manufactured by the Dorr Co., Stamford, Conn.

F. M. LEWIS, Member AIME, is Superintendent of Concentration, Tennessee Copper Co., Copperhill, Tenn. E. C. JOHNSON is Sales Representative with the Dorr Co., Stamford, Conn.

Discussion on this paper, TP 3803B, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Feb. 12, 1954. New York Meeting, February 1954.

admittedly undersized, but the increase in grinding efficiency, Table I, when the cyclone was placed in operation is of interest.

Table I. Improvement in Regrind Circuit After Change from Undersized Classifier to 24-In. Cyclone

Machine	Period of Time Over Which Averages Were Made	Raw Feed		Tons −200 Mesh Per Day	Ball Mill Hp	HP Per Ton of −200 Mesh Made
		−200 Mesh, Pct	TPD			
Classifier	Jan-Apr 1951	63.9	1286	104	155	1.49
Cyclone	Year 1952	65.8	1286	177	131	0.74

Screen Analysis

Mesh	Average Feed	Cyclone		Classifier	
		Overflow	Underflow	Overflow	Sands
100	1.3	0.2	2.9	1.6	13.5
150	18.9	4.9	37.1	10.8	56.3
200	37.4	17.0	63.9	26.4	79.2
−200	62.6	83.0	36.1	73.6	20.8
Solids, Pct	50.6	40.9	73.3	41.1	82.7

The figure which draws immediate attention is the reduction from 1.49 to 0.74 hp consumed per ton of −200 mesh material produced; the 50 pct reduction speaks well for the proper sizing of classifiers in circuits such as this. The accompanying 70 pct increase in the quantity of −200 mesh material produced with the 15.5 pct decrease in total ball mill horsepower bears this out.

The power reduction shown in Table I can be attributed to several factors. With the undersized classifier in the circuit it was not possible to build up the necessary circulating load of sands to the mill. This caused the density in the mill to be low and at the same time increased detention time. The increased detention time was primarily a metallurgical factor in that it caused the mill to go acid, greatly affecting the copper recovery. Attempts made to correct these conditions included sending the raw feed directly to the mill and increasing the ball load. (The above classifier data were collected after the ball load was increased.) Installation of the cyclone with the subsequent increase in circulating load and grinding efficiency permitted a reduction in ball load which accounts for the decrease in power.

Comparing the screen analyses of the two indicates that the bowl classifier provided a cleaner sand. Decreasing the diameter of the cyclone apex valve would have increased the underflow percent solids,

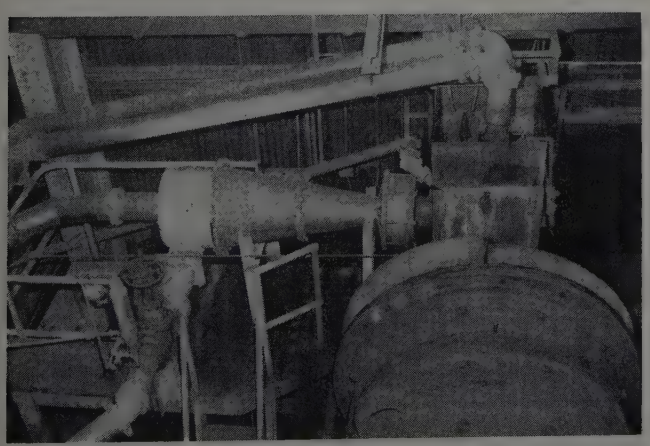


Fig. 2—View of cyclone installation at Tennessee Copper Co., Copperhill, Tenn.

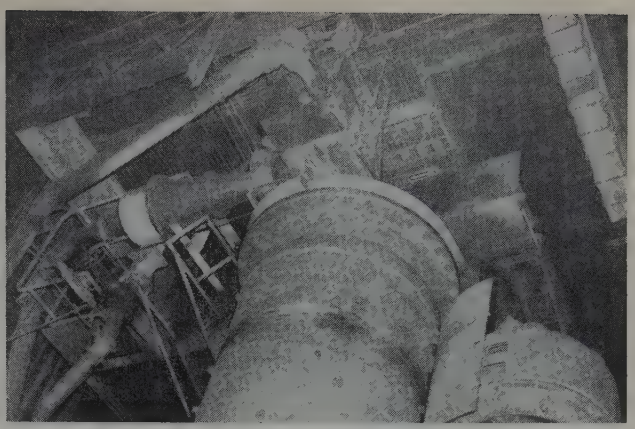


Fig. 3—View of cyclone classifier, taken from overhead crane, showing small space occupied by unit.

making it possible to approach the classifier rake product in this respect.

Operating the cyclone, in this instance with the apex valve wide open to provide the greatest possible circulating load, gave a sands product higher in −200 mesh material but at the same time provided an overflow which was believed to function best in the flotation circuit. Production of a grind which provided the greatest possible yields in this flotation circuit was, after all, the goal.

Operation Costs and Wear

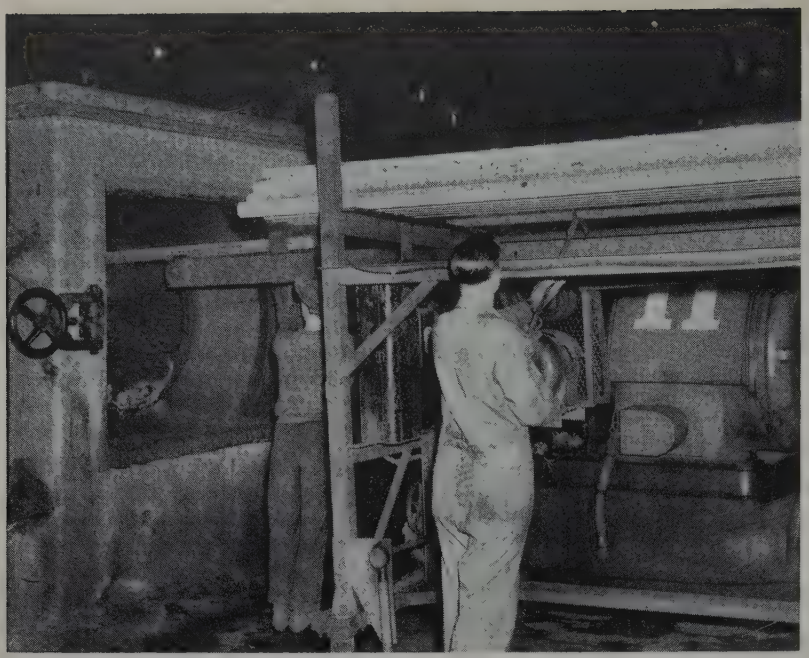
Pumping 497 gpm of a 50.6 pct solids slurry through a device such as this liquid-solid cyclone will, of course, produce wear on at least some of its parts. As originally supplied, the cyclone was completely rubber-lined, except for the feed chamber. Since then this section has also been rubber-lined. In this operation the vortex finder has been the part that has shown the most wear. Life of a vortex finder has varied from 100 to 150 days. The feed and conical sections have an estimated life of three years. The apex valve shows no sign of wear. Using only 5 psi feed pressure at the cyclone helps considerably in keeping the cyclone maintenance low.

Table II compares operating costs of the liquid-

Table II. Comparative Daily Costs of Operating and Maintaining a 24-In. Cyclone and a Sedimentation-Type Classifier

	Cost, $
Cyclone	
Maintenance: pump	
Impellors, suction and gland side liners, casing plate	2.24
Labor	0.04
Cyclone: vortex, head and cone, apex valve	0.41
Power, $0.0044 per kw-hr	0.83
Total	3.52 per day
Classifier	
Maintenance: rakes, screw conveyor, scoop feeder	1.21
Power	
Scoop feeder	0.39
Classifier	0.32
Total	1.92 per day

solid cyclone and sedimentation-type classifier performing in the closed-circuit described here. Although the cyclone installation costs 83 pct more to operate than the sedimentation-type unit, in many cases the difference in initial investment will justify use of the cone. Furthermore, in a plant that is expanding its capacity or changing to a closed-circuit operation the cyclone offers the advantage of being adaptable to existing conditions. It requires less floor space and in many instances can be mounted directly over the grinding mill.



Methods of Charging Rods and Balls Into Grinding Mills

Fig. 1—The rod charging machine used at International Nickel's Copper Cliff concentrator loads 34 rod mills three times a week. With this machine three men accomplish in less than 2 hr a task that once required eight men working 4 hr.

by Oscar Johnson

The industry has been canvassed for information on methods of handling grinding steel at milling plants, an important subject overlooked in most descriptive articles on milling plant arrangements. The data collected are offered in this paper as a guide to designers and planners who have loading and storage problems to solve.

WITH capacities of milling plants expanding from a few hundred tons per day to many thousand, replenishment of grinding media is an important phase of mill operation. Information on this aspect of milling procedure, solicited from several mining companies, is summarized here.

At **International Nickel's** Copper Cliff concentrator the normally strenuous job of changing rods is considerably simplified by the use of the Garrow rod charging machine, Fig. 1. The overhead crane, Fig. 2, brings a bundle of rods, weighing four to a ton, to the loading platform of the machine, positioned in front of the mill. The rods roll one by one down the slight incline of the platform onto a conveyor which sends them into the mill.

A modified version of the Garrow machine, Fig. 3, is used at the Federal mill of St. Joseph Lead Co. An overhead crane places the portable steel structure in position in front of the discharge end of any one of the five 9x12-ft rod mills. It then raises a bundle of about twenty 3-in. rods to the discharge platform of the charging machine. The rods are 11 ft 7 in. long and weigh about 278 lb. By tripping a lever the operator allows one rod at a time to roll onto the inclined rollers. A slight push by the operator sends the rod smoothly into the mill.

O. JOHNSON, Member AIME, is Chairman of the Board, The Mine and Smelter Supply Co., Denver.

Discussion on this paper, TP 3808B, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Jan. 13, 1954. New York Meeting, February 1954.

The **Union Miniére du Haut Katanga** rod charging machine, Fig. 4, is mounted on a pair of wheels at the forward end with swiveling castors at the back for steering it to position for use or storage. Wheels are equipped with locking brakes to maintain the machine in position while it delivers rods to the mill.

The device is made in two parts. The first consists of the base, which carries the supporting wheels, grooved rollers, and drive. The other part serves as the rod deck, which can carry up to six 3-in. x 12-ft rods. A short boom built integral with the base reaches through the mill trunnion and delivers the rods into the mill barrel.

Three grooved guide rollers are provided to receive the rods from the sloping charging deck. One rod at a time is fed to the rollers and thus delivered into the mill. Two of the rollers are driven by 1½-hp motor through a *V*-belt drive. The third roller is driven off the first by a single *V* belt in a groove below the rod groove and on the same center line. Rods are delivered to the charger by an overhead crane in bundles of three to six as needed.

The power is supplied by cables plugged into power outlets, suitably located near the rod mill discharge end.

The **Oliver Iron Mining Co.** uses a rod charger, Fig. 5, suspended from a monorail by cables. It is constructed of structural steel and has two decks, each capable of carrying five rods of 4-in. maximum diameter. The rods ride in grooved free-turning rollers. Legs extending down from the mill end of the mechanism act as supports when the unit is ex-

tended into the discharge trunnion liner. When the mechanism is placed in the trunnion, the projecting end of the charger is elevated by a chain-and-sprocket lifting block providing slope to the charger. A lever for each deck allows the operator to release five rods at a time and gravity carries them into the mill interior. It is found that pushing the protruding ends away from the axial center line of the mill will carry the rods all the way into the mill, thus preventing their tangling by catching in the space between the rods which constitute the normal charge. Only one man on the machine is required, and 10 rods can be put into the mill in about 1 min. The rod mill is stopped momentarily.

At **Chile Exploration Co.** rods are brought in by railroad flat car and spotted beneath a monorail, which carries an electric hoist with overhead cab control and has two 16x24-in. rod-handling magnets. The monorail extends over the rod storage bins and the rod charging car. One man operates the hoist and another guides the rods to keep them parallel. Three 700-ton storage bins inside the concentrator and additional bins outside are served by the same monorail system.

Rods are charged by a battery-propelled machine, Fig. 6, traveling on 84-in. gage rails behind the mills. They are dropped one at a time on a spinning roll which thrusts them into the mills; a telescopic extension prevents their dropping on the discharge trunnion. Only two men are necessary at the charging machine, but five are needed to operate the heavy plug doors, which are opened and shut while the mills are running. Mills are shut down for about 3 min every other day to charge 15 to 18 rods. A printomatic track scale is available to weigh charges.

Balls are received in steel drums and dumped from these into one of six 400-ton storage pits. A floor-controlled electric hoist with a magnet of 45-in. diam is suspended from a monorail running over the pits and the car charging hopper. The magnet delivers the balls from the pits to the hopper, which discharges through a gate into a ball charging car. This car, of 2-ton capacity, is driven by a small battery locomotive on a 24-in. gage track in front of the mills. At each mill a chute delivers balls to the drum feeder through the feed launder. The car has a gate and a hopper bottom which delivers balls to the chute. One man working part time weighs the charge on a printomatic track scale. By means of a gate on each mill chute, a charge for two days is placed the same day.

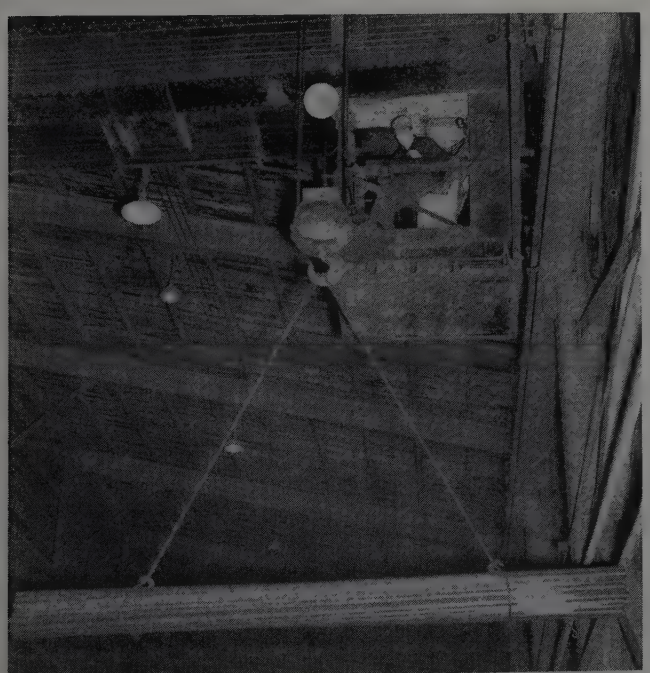


Fig. 2—The overhead crane at Copper Cliff brings a bundle of rods from the empty stock in the concentrator. Rods weigh four to a ton.

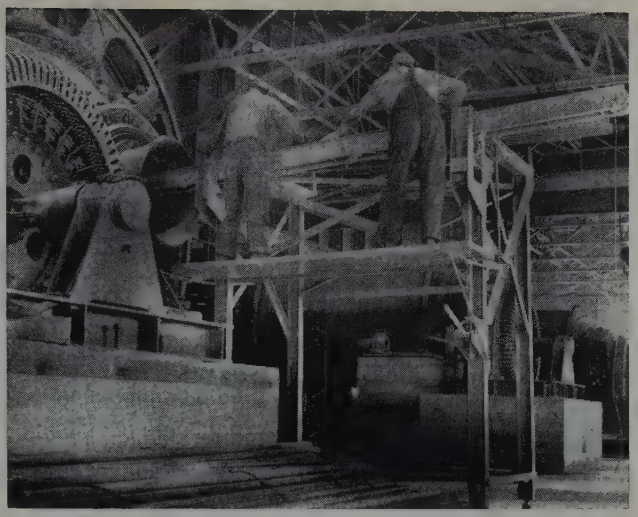


Fig. 3—The above rodding device at the Federal mill, St. Joseph Lead Co., is a modified version of the one used by International Nickel Co. in Canada.

When a similar method is used to charge the regrind mills, balls fed through the drum feeder are thrown out by the scoop. Because of this fault in scoop design, regrind mills are loaded through the shell doors about twice a month. Balls are fed to the mills by a gate-controlled hopper suspended from a crane hook. One and a half hours are necessary for each charge.

At **Cananea Consolidated Copper Co.** balls for the eight 10x10-ft Marcy ball mills are received in railroad box cars, 60 tons to the car. At the unloading point the railroad track is above the general yard level and the balls are rolled and pushed from the cars into a chute leading to a 10-ton end-dump truck, which delivers to the concentrator ball bins 0.6 miles distant.

Walls of the 350-ton capacity concrete bins are 1 ft thick and floor slabs 2½ ft thick. Suspended from a trolley on a 10-in. I-beam is a Yale Cable King hoist of 2-ton capacity. The hoist carries a 39-in. high intensity lifting magnet using 230 v dc. The trolley is operated by a dc motor of 7½ hp. A load of balls picked up by the magnet from any one of the four bins is dropped into a hopper, which discharges into a box or car body mounted on a trailer hauled by an electric motor, both running on a 24-in. gage track. The track is supported on a concrete shelf which is part of the common wall of the concentrator and the fine ore bin.

The trailer truck of the special electric wheel tractor, Fig. 7, has a simple pin coupling on each end to connect it with a similar coupling on the other end of the tractor. The box holding the balls, which is mounted on the trailer, is constructed of ⅜-in. plate with angle reinforcement at the corners around the top. On the track there is a dial scale of 5000-kg capacity, equipped with a printing device.

At each of the eight grinding sections balls are delivered from the car to the feeder drum of the mill by a steel chute, which is lined with discarded conveyor belting to decrease wear and eliminate

Fig. 4—The rod charging machine shown above, Union Miniere du Haut Katanga, is equipped with swiveling castors to position it for use or for storage.

noise. It is flared at the upper end and extends under a lip at the gate of the ball box on the trailer truck. The chute is 8 ft 8¾ in. long and 18 in. deep, narrowing from 2 ft to 15 in. wide and sloping at 3½ in. per ft.

At the end of this launder there is a downspout 7 ft 3 in. long and 12x15 in. on the inside. The bottom is made of ½-in. plate. Should there be a choke, which seldom occurs, one side of this spout is removable. From the side and at the bottom of the downspout a launder 15 in. long, 12 in. wide, and 10½ in. deep, with a slope of 2½ in. per ft, delivers the charge to a box which receives the ore feed for the section. The box discharges down the feeder spout into the feeder drum of the mill. To prevent possible choking, the ore feed is stopped for the few minutes necessary to charge the balls.

If possible the daily charge, which is governed by the operating time of each mill, is a little less the actual wear rather than more. From past experience, 900 kilos is considered the charge necessary for a 10x10-ft Marcy mill in a 24-hr day. When a mill has run 24 hr the preceding day, as is usual, it receives 900 kilos. If the time is less, the charge is adjusted accordingly. If on occasional inspection the superintendent considers the ball level too high or too low, extra charges are put on the daily sheet or the regular charge is omitted. Often an entire month passes without any of these adjustments. Ball levels are maintained 6 to 8 in. below the center line of the mill.

In the supply department ball stocks are carried by bins as well as by order numbers. When a bin is empty a cutoff is made and any shortage paid for immediately. If there is an average it is absorbed by current operations.

Since one man can charge the balls in less than 3 hr, a daily average of 3 hr should take care of the regular charges and any extras. Unloading a 60-ton car into a truck requires the labor of the truck driver and three men for 4 hr.

Guy H. Ruggles, who describes these operations at Cananea, considers this the best ball charging system he has ever seen, except that he prefers delivery to bins by railroad cars rather than by truck haulage. The system was designed in the New York office of Anaconda Copper Mining Co.

In addition to the eight Marcy mills at Cananea there are three Allis-Chalmers trunnion discharge mills, 6½x12 ft, for regrinding concentrates. These are charged with 1-in. diam balls plus the scrap which passes the grates of the Marcy mills and is caught in the discharge boxes and launders. Balls are unloaded from the box cars at the same location, whatever the size, but the 1-in. balls are carried by wheelbarrow from the cars to the loading chute. A crew of three men and the truck driver can unload a car in about 12 hr. The truck dumps the small balls into a low wooden pen on the floor. They are then transferred to one of three wooden bins by a 39-in. magnet suspended from the 10-ton crane that serves the regrind mills. A reelite on the crane handles the power cable to the magnet.

For charging the regrind mills there is a steel box 4 ft square, 4 ft 4 in. deep at the front side and 3 ft 3 in. deep at the back, with a spout and a lever gate in front and angle iron legs. After the balls are put into this box by the magnet it is placed in front of the mill, with the spout, which is 13 in. wide, projecting into the combination feeder of the mill scoop. The box is hoppered both ways toward the gate, and with some assistance the balls flow into the feeder when the gate is opened.

Charging is regulated according to the ammeter reading of the 300-hp 2300-v motors. If the ammeter reading is higher than 42, which has proved the most satisfactory, the balls are thrown out of the mill and into the sand wheel on the mill discharge.

About once a week two men working on the floor and one in the crane charge all regrind mills in 3 hr.

E. G. Lewis of **Phelps Dodge Corp.** writes that mills at the Ajo, Ariz., plant are replenished through the discharge heads:

"Grinding balls are delivered to the mill by rail in gondola cars. The cars are switched into the mill building where storage bins are located on both

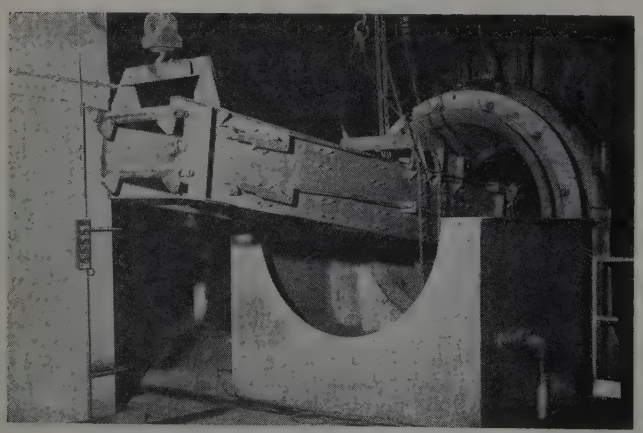


Fig. 5—This rod charger, Oliver Iron Mining Co., is suspended by cable from a monorail.

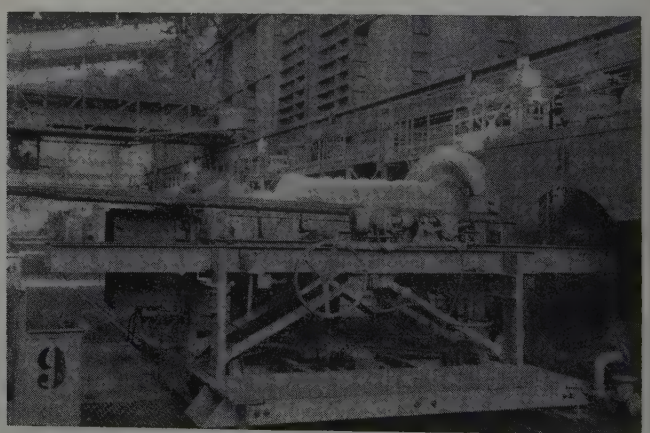


Fig. 6—The battery-propelled rod charging machine at Chile Exploration Co. travels behind the mills on 84-in. gage rails.

sides of the track. The balls are unloaded from the cars with an overhead crane and electric magnet, two men being required for this operation, [a] craneman and a man on the magnet switch. The two storage bins are divided into three compartments each for segregation of sizes. A gate is provided at the bottom of each compartment for drawing balls from the bins. These gates are operated by rack and pinion.

"Steel boats [Fig. 8] carried by an overhead crane are used in balling the mills. The boats have a capacity of 5000 pounds and have a swinging gate and spout at one end for discharging the balls into the mills. In the ball charging operation the loaded boat suspended from the crane is anchored to the floor by means of a chain attached beneath the spout, which rests on the mill discharge box. A short portable steel launder is used as a runway for the balls from the boat spout to the opening in the center of the discharge head of the mill. This opening has a widening taper into the mill. As the boat is unloaded the rear end is gradually raised by the crane to allow easy flow of the balls from the boat.

"The balling crew consists of four men including the craneman. One man loads the boats from the storage bins while the other two charge the mills. The average time cycle for balling each mill is fifteen minutes. Balls are put into the mills while they are in operation."

At **Climax Molybdenum Co.**, according to Frank Coolbaugh, Resident Manager, two separate methods are used to unload and store the balls. Consistently accurate results have been obtained for a number of years. Figs. 9 and 10 illustrate the handling methods used for No. 2 mill and for No. 3 and 4 mills.

In the No. 2 mill, comprised of eight mill, classifier, and flotation units, balls are stored in heavy wooden bins located between the ore bins and outside the milling section. Because the bins are not situated advantageously with respect to the railroad, balls are unloaded by trucks, discharged into hoppers on high ground above the bins, and fed by gravity to the bins through open launders. One man transfers the correct number of balls every day from the bins to individual hoppers near each scoop box. Balls run by gravity from bins to hoppers through 4-in. standard pipe.

Because the number of bins permits storing a wide range of ball sizes, efficient mixing and testing are possible. Each bin can be piped to feed one to four mill hoppers (not shown in Fig. 9).

"In the new No. 3 and 4 mills with a total of six units," writes Mr. Coolbaugh, "ball storage has been

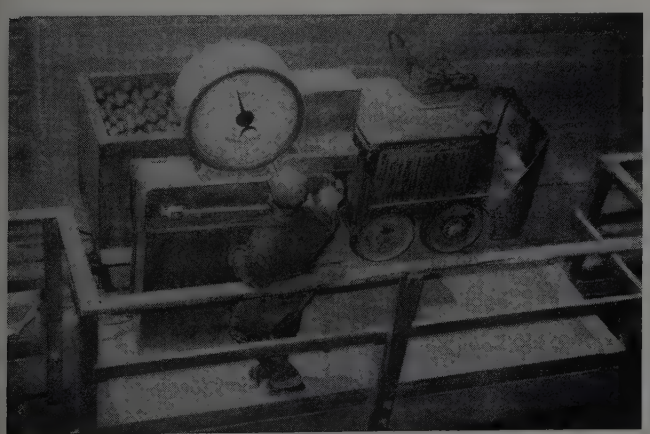


Fig. 7—The electric-wheel tractor with trailer truck, Cananea Consolidated Copper Co.

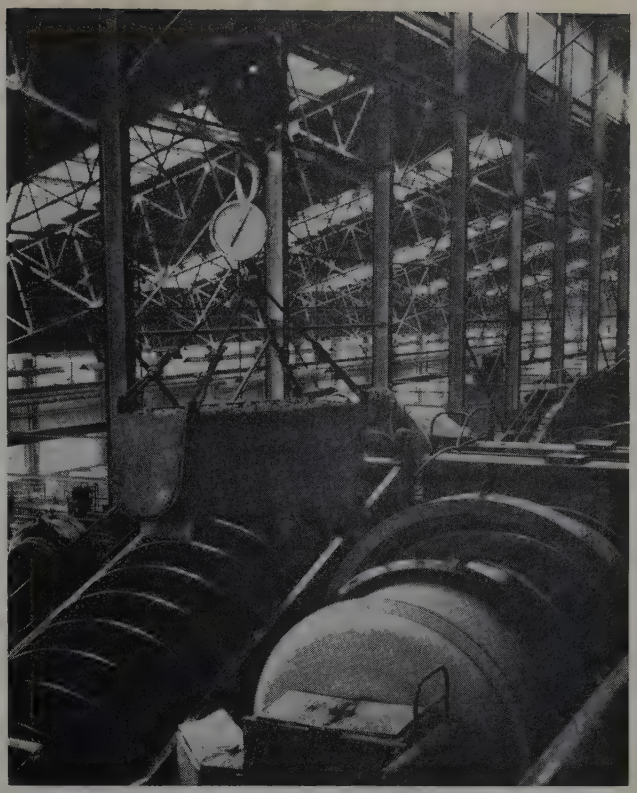


Fig. 8—Steel boat used in balling mills at the Phelps Dodge concentrator at Ajo, Ariz.

provided inside the mill building in one large divided concrete bin with a capacity of approximately 1500 tons of balls for six different sizes and makes. An overhead crane with magnet quickly unloads from railroad gondola cars or trucks into the storage bins. This magnet also charges the balls to be fed into a transfer bucket for each individual mill. The transfer bucket is picked up by the large mill crane and discharged into ball hoppers located close to the inspection door on the scoop boxes."

One man handles unloading into storage bins, charging from bins to hopper and from hopper to transfer bucket, and unloading into individual hoppers next to the mills. He is assisted by the crane operator when the transfer bucket is moved from one mill to the next.

In all mills the balls are dropped one at a time directly into the scoops while the mill is running. Day-shift operators can fit this duty into their schedules whenever it is most convenient.

At **Anaconda Copper Mining Co.** modernization plans are not yet completed. In the meantime an overhead crane is being used to lift grinding rods from warehouse stock to the structural steel carriage, which is mounted on wheels and moved by overhead crane. The carriage and the rods are placed directly in front of the 9x12-ft rod mills, and by means of a ratchet and lever, individual rods are placed on live rolls driven by a motor and V-belts. The action of the live rolls throws the rods through the large opening of the Marcy rod mill directly on top of the rod charge.

Small balls are used in the secondary ball mills which operate on a discharge from the rod mills. These balls are loaded in steel containers, elevated by traveling crane, and dumped as required into the ball mill feeders.

As reported by C. G. McLachlan, rod handling at **Quemont Mining Corp.** presents little difficulty, since only one Marcy rod mill, 9 ft 5 in. by 12 ft, is

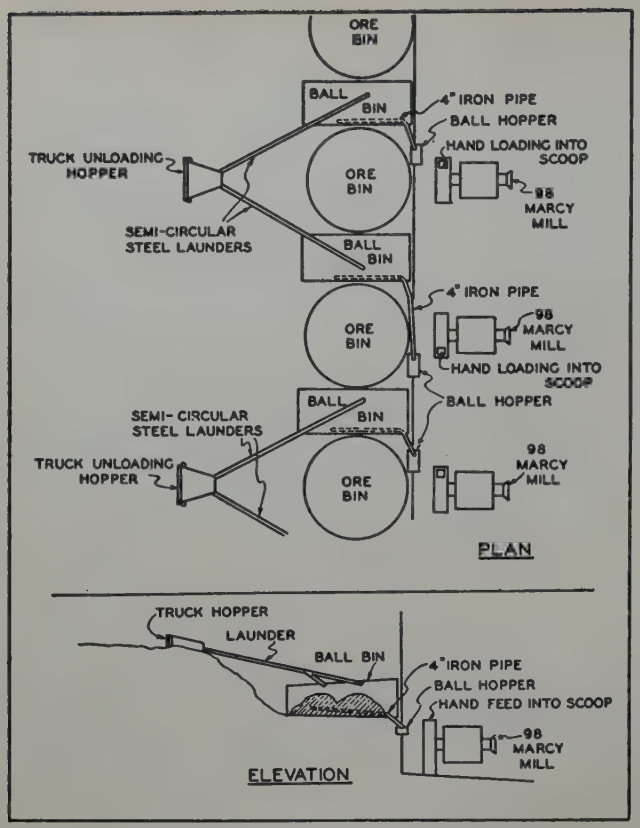


Fig. 9—Elevation and plan of No. 2 ball mill at Climax Molybdenum Co.

operated. The mill is charged with rods 11 ft 8 in. long, of 3½ in. diam, weighing about 390 lb each. Strapped together in units of ten, these are brought in by rail to the Quemont siding, transferred by crane to a truck, and unloaded by crane at the main stockpile, which is located outside the concentrator building near the rod mill grinding bay. There is a smaller stockpile on the ground floor inside the building, approximately 10 ft below the rod mill work floor. Rods are moved from the ground floor to portable racks on the work floor by a 20-ton rod mill service crane.

Twice weekly the mill is loaded with 18 to 21 rods, charged manually one at a time from a portable set of rollers mounted on a light "A" frame and inserted into the discharge trunnion. Seven men working approximately 20 min are required to charge the mill. Total *down* time due to rod loading is about 40 min per week.

At **Quemont Mining Corp.** all grinding balls are delivered by truck to the concentrator and dumped into bins through loading chutes outside the building. Each of the three bins, of laminated wood construction, holds 60 to 70 tons of balls. The bins discharge through small chutes which may be raised or lowered to control the flow of grinding media.

Each of the five 9x12 ball mills is charged daily, three with 2-in. steel and two with 1½-in. steel. The 5 tons of grinding media are charged in 1 hr by a crane operator and assistant.

A 15-ton crane services the mills, which are installed parallel to each other. A loading bucket of about 1-ton capacity, Fig. 11, is lowered to the discharge chute of the appropriate bin and filled. The bucket is then carried to the discharge end of a ball mill and lowered until its spout meets the cut-away section of a 10-in. pipe, which delivers balls into the mill. All mills are fitted with reverse spiral discharge trunnion liners to facilitate moving the steel into the mills.

At the **Phelps Dodge** plant at Morenci a 3-months' supply of grinding balls is kept on hand, since Morenci is far from ball manufacturing centers and would be seriously affected by a steel strike or a railroad strike. There are three storage areas, a large primary storage bin outside the southwest corner of the concentrator building and two smaller rationing bins at each end of the building. The primary bin, of reinforced concrete 18 in. thick, has four compartments partitioned by walls of the same material and thickness. The inside walls are covered with used conveyor belting to prevent chipping and cracking of the concrete when loads are dropped into the bins. Total capacity of the primary bin is well over 3000 tons.

The two rationing bins are located above the loading points for the ball cars. One is divided into four compartments, each with a capacity of 120 tons, and the other into three compartments, each with a capacity of 130 tons. Incoming carloads are diverted to the rationing bins whenever possible; however, if they arrive too close together the balls are unloaded at the main storage bin and transferred as needed.

Balls are transferred from the cars to the main storage bin by a moto-crane with an electro-magnet capable of swinging a ton of balls per load. Unloading time varies from 1 to 1½ hr, depending on conditions existing at the point where the car is spotted. The time is about the same for reloading a flat car when the rationing bins are replenished from the main storage bin. Cars at the overhead bins are

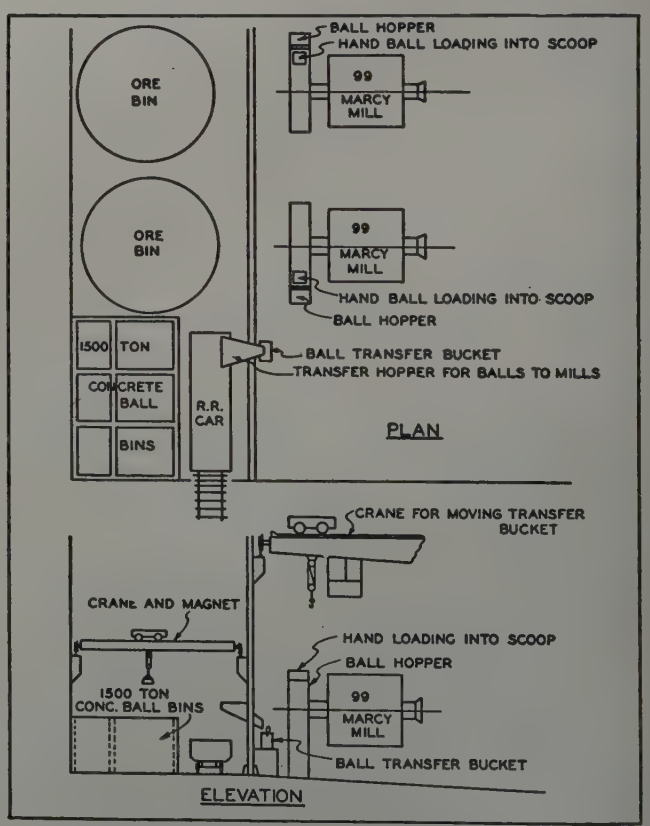


Fig. 10—Elevation and plan of No. 3 and 4 ball mills at Climax Molybdenum Co.

unloaded by a magnet on a traveling crane in the mill itself, the time required averaging 2 hr.

A 24-in. gage railroad runs from one end of the concentrator to the other, passing the mill scoop boxes. Balls are discharged from the overhead bins by gravity flow over a counterbalanced gate spout to a trailer drawn by a battery-operated locomotive. The trailer is drawn over a scale where the load is weighed and recorded. There are two locomotives, each operated by one man. A third man weighs the load, records the weight, and specifies the weights desired for each mill.

When balls are transferred from the cars to the mills, the trailer is spotted between the central point of the mill scoop housing and the sand discharge end of the classifier. The sliding gate on the side of the trailer is opened and the balls roll out over a short guide spout and through a rubber-lined chute to the specially designed drum feeder section of the grinding mill scoop, which takes them into the mill. Charging 28 mills with a ton of balls each requires about 2 hr. The quantity rationed to each mill is determined according to a daily consumption rate adjusted periodically after inspection of the height of ball load. Ball loads are kept to within 4 in. above or below the center line.

Smaller operations are carried out by the **Idarado Mining Co.** of Ouray and Telluride, Colo. Incoming shipments of balls are hauled from the railhead by standard dump trucks to rectangular log cribs built on the ground. New balls, added each shift to compensate for wear, are transported from cribs to a small storage bin near the ball mills as needed. This is accomplished by operating personnel whenever time is available. Balls are carried on the ore feed belt to the mill equipped with a combination feeder. They are dropped into the rotating scoop of the mill equipped with a conventional feeder, having been carried by electric hoist to the top of the scoop box in a steel bucket of 500-lb capacity. This is also done by operating personnel.

When a ball mill is to be relined the full charge of 30,000 lb is dumped on the floor underneath the mill. Three or four men then load it into the specially designed bucket (2000-lb capacity) of a Krane-Kar equipped with a swinging and hoisting boom. The bucket is hoisted to rest on a hopper placed in the manhole and the balls are dumped through the drop bottom. Reloading time is 2 hr.

Procedure is similar at the Telluride mill, except that the bucket for loading balls back in the mill is patterned after the drag-line type. An electric Anaconda hoist is equipped with a Sullivan tugger which holds the bucket away from the mill while a load of balls is being hoisted. When the bucket is in the dumping position the tugger is slacked off, allowing the front end to drop.

The ball bucket, 40 in. long, is 24 in. wide and 18 in. deep in back and 12 in. wide and 9 in. deep in front. It is manually loaded with shovels by four men working in two-man shifts. Shifts are rotated after each man has mucked two buckets, enabling the men to work at top speed. Reloading time ranges from 1 hr to 1 hr 35 min.

At **Waite Amulet Mines Ltd.**, Noranda, Quebec, grinding rods are charged manually to a 9x11½-ft mill. Clearance between the trommel screen of the mill and the wall of the mill building is 4 in. more than the length of the rod used, but the axis of the mill passes through the center of one of the two 5-ft servicing doors, which is opened when the rods are

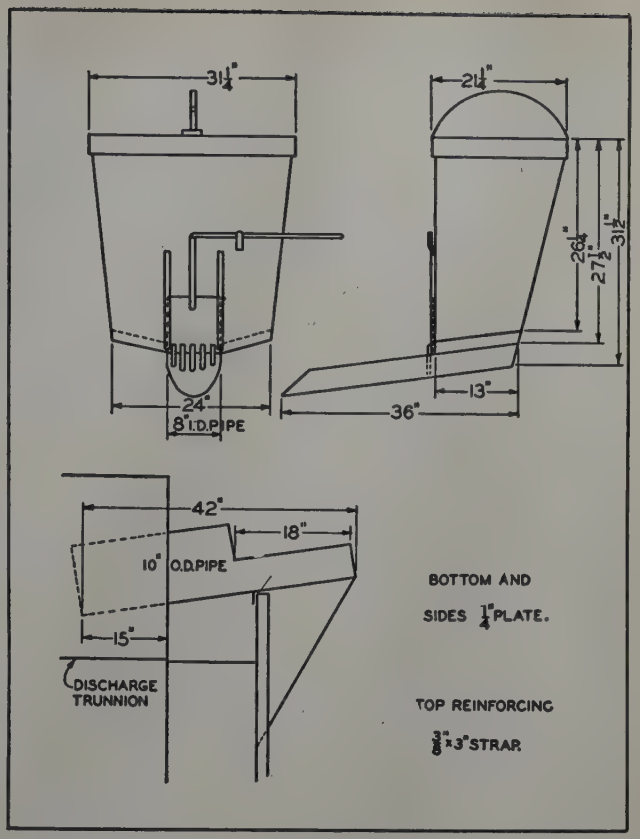


Fig. 11—Ball charging equipment used at Quemont Mining Corp. Bucket spout and loading pipe slope 2 in. per ft.

charged once a week. A surface gang of six men with foreman charges 30 rods in about 20 min, using the following procedure: 1—A roller is placed at the discharge end of the mill, and a saw horse on the floor 7 ft from the roller. 2—Two men rest a half pipe on the roller and the saw horse, while four men place a rod in the pipe, the outer end at stop *a* and the other end extending into the mill. 3—The two men handling the half pipe raise its outer end and, after the saw horse is removed, lower the pipe slightly and push it into the mill until stop *b* reaches the roller. The pipe is rotated through 180° to discharge the rod. It is then withdrawn and the procedure outlined in step 2 is repeated.

Stops *a* and *b* are located to spot the rod correctly in the mill. Prior to their use there was a tendency to push some rods into the feed screw or to leave them in the discharge opening. It is not difficult to drive a rod from the discharge opening into the mill, but there is considerable delay when rods are placed upon others which have been pushed into the feed trunnion.

Conclusion

By means of mechanical operation, large tonnage plants have solved much of the problem of loading, but despite the known benefits of reducing time losses many plants find it difficult to justify the expenditure required to improve methods. The economies of each method of operation should be evaluated carefully.

Acknowledgment

The author wishes to thank the men, and their companies, who so kindly supplied the information on what is necessary to keep the long line of rod and ball mills grinding at top efficiency.

Sedimentary Rocks at Cananea, Sonora, Mexico, And Tentative Correlation with the Sections at Bisbee And the Swisshelm Mountains, Arizona

by Roland B. Mulchay and J. Ruben Velasco

CANANEA has long been recognized as a remarkable field for geologic study. The copper deposits and rocks of the district have been described by many geologists and engineers, but only the most general correlations have been made between Cananea sedimentary rocks and other known sedimentary sections in the southwestern United States and northern Mexico. The present paper describes the Cananea sediments in greater detail than has been done before and attempts to fit the Cananea sedimentary section more closely into the geologic time table. The lack of well-preserved fossils has made it difficult to date the sediments accurately in geologic time, but it is possible to make tentative correlations between the Cananea sediments and the southeastern Arizona sections, based largely upon lithology and general position in the geologic column. It appears that sedimentation at Cananea and Bisbee may have been closely similar during Paleozoic time. Even such generalized correlations, however, may be subject to considerable modification in the future. The present study has led to the recognition of other problems of age and mineralization relationships in the Cananea district.

Cananea is located in the north-central part of the state of Sonora, Mexico, at an elevation of 5270 ft. It is about 135 miles northeast of Hermosillo, the state capital, and 25 miles south of the international boundary. By road Cananea is 40 miles from the twin towns of Naco, Ariz., and Naco, Sonora, and about 50 miles from Bisbee, Ariz. It is served by the Nogales-Naco branch of the railroad, F.C. Pacifico, and is connected with Chihuahua and Mexico City by the Aeronaves airline.

The headwaters of three rivers flowing to the Gulf of California are located in the Cananea Mountains: the San Pedro River, flowing to the north; the Sonora River, flowing south and west; and the Magdalena River, flowing west. Elenita Mountain, the highest point in the district, has an elevation of 8140 ft. The Cananea Mountains extend in a series of north-south to northwest-southeast spurs and ridges and are surrounded by gently sloping gravel plains. The mineralized area, lying across the southern and central parts of the range, is about 6 miles long and at most 2 miles wide. Elevations at the mines vary from 5300 ft at Cananea-Duluth mine at the southeast end of the district to between 6000 and 7000 ft at the west end of the mineralized area at Puertecitos-Elenita mines. Principal production has been from the intensely mineralized and altered area of Capote Basin in the central part of the district and the immediately surrounding area to the southeast. The district has produced over 2 billion lb of copper, substantial molybdenum, and minor amounts of lead, zinc, silver, and gold. Total production through 1949 is estimated at more than $300 million.

In 1900 large-scale development was started at Cananea by W. C. Greene. Until World War II only high-grade ores were exploited; low-grade ores were extracted after the installation of a large concentrator in the early 1940's, and subsequent operations have been based upon mining and processing ores containing less than 1.0 pct copper from open-pit and underground workings. Mining and concentration of such low-grade ores, however, are made possible only by continued high copper prices, and active exploration for high-grade orebodies has been continued throughout the important mineralized areas.

General Geology

Study of the involved rock pattern at Cananea has indicated a complex geologic history for the district. Widespread alteration and mineralization have masked many of the salient features and have led to widely varying geologic interpretations over the years. Further work will probably disclose new information which will modify current beliefs.

At Cananea a conformable series of sediments of probable Paleozoic age was deposited on an unknown basement. Following Paleozoic time there was an extended period of erosion common to many districts in the southwestern U. S., and there is no present evidence of marine sedimentation at Cananea after the Paleozoic. The eroded surface was eventually covered with a great thickness of extrusive volcanic rocks. The entire series of sediments and volcanic rocks was later intruded by a variety of deep-seated igneous rocks. These included the Cananea granite, the Cuitaca granodiorite, the El Torre syenite, the Tinaja diorite, the Campana diabase and gabbro and the Colorada rhyolite quartz porphyry. Faulting of early age, probably prior to the deposition of the volcanic rocks, may have been responsible for the present position of some of the intrusive rock masses. In the Capote mine on the third and fourth levels the northwest-striking Ricketts fault zone, with apparent offset of about 800 ft, has been sealed by a dike-like mass of Cananea granite which gradually increases in size with depth. In lower levels of the mine the granite forms a large southeast-plunging mass generally following the course of the Ricketts zone. The granite is not known southeast of the Capote-Oversight mine areas and the Ricketts fault does not appear in the volcanics southeast of Capote Basin, but several plugs of Colorada quartz porphyry cut the volcanics along the assumed general southeast trend of the Ricketts zone. These porphyritic intrusives may be the up-

R. B. MULCHAY, and J. R. VELASCO, Members AIME, are, respectively, Exploration Geologist, Anaconda Copper Mining Co., and Chief Geologist, Cananea Consolidated Copper Co., Cananea, Sonora, Mexico.

Discussion on this paper, TP 3790I, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, July 3, 1952. Mexico City Meeting, October 1951.

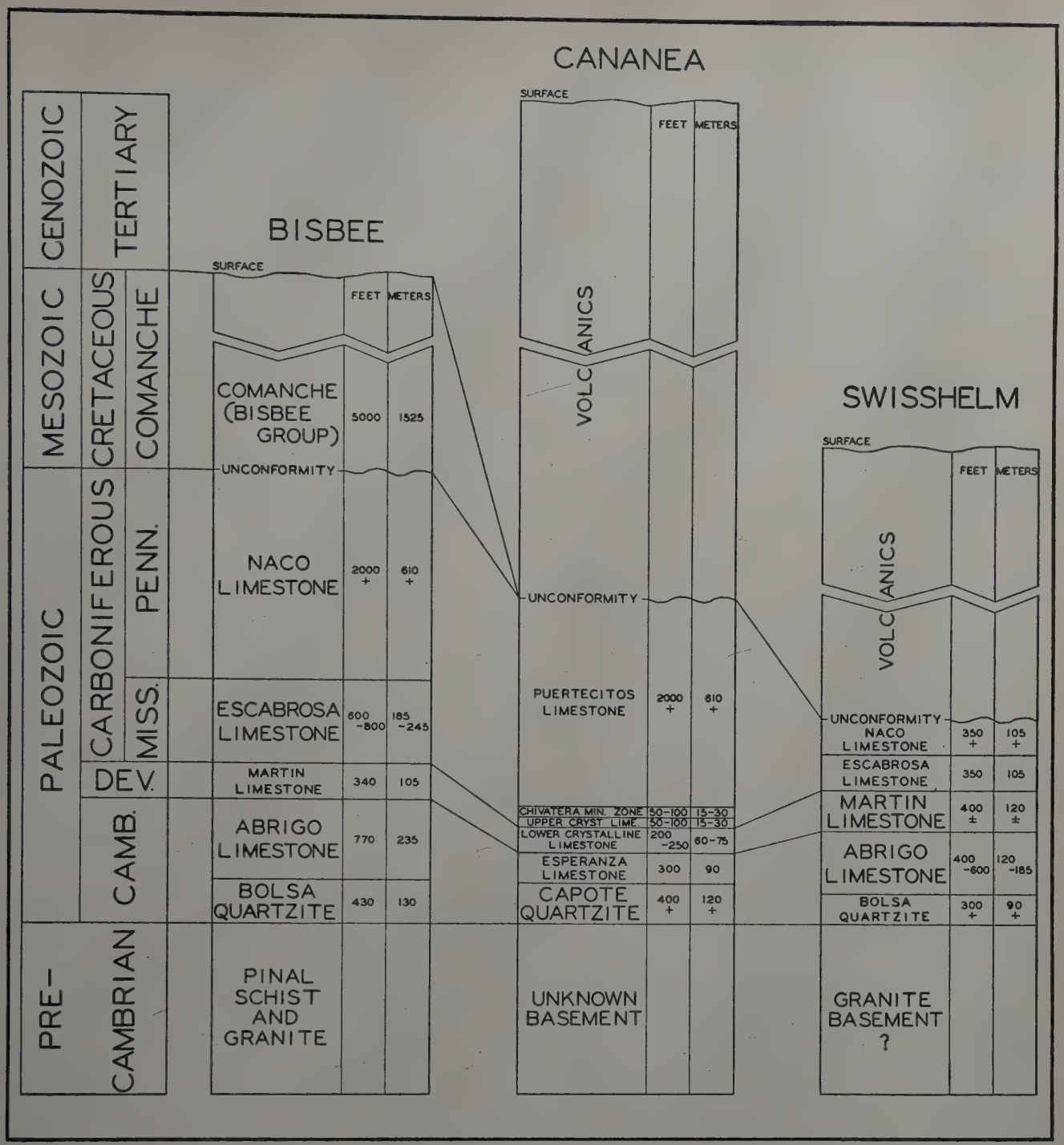


Fig. 1—Comparative stratigraphic columns for Cananea, Bisbee, and the Swisshelm Mountains.

ward expression of a batholithic mass of granite deeply buried beneath the volcanic rocks in the southeast end of the district.

Mineralization occurred in several surges. Sulphide minerals in economic amounts, often locally enriched, were deposited in the sediments, volcanic rocks, and quartz porphyry. Throughout the district upper limestone beds were replaced by garnet. The intimate relations of the garnet and some of the ore minerals suggest that at least part of the widespread garnetization was caused by hydrothermal solutions rather than by limited contact metamorphic effects. Intense sericitization of the volcanic rocks and quartz porphyry was directly related to strong mineralizing activity.

Cananea is lacking in prominent vein structures. Most of the economic mineral deposits are localized in steep-dipping breccia pipes, in disseminated deposits in the volcanic rocks and quartz porphyry, and in replacement deposits in the sediments. Much of the copper ore mined during the past 25 years appears to have been closely related to the quartz porphyry intrusives. After detailed study of the contacts of these intrusives with the volcanic rocks many of the known low-grade ore areas have been enlarged. Mineralized breccia pipes, some of which do not reach surface, have formed feeders for the disseminated minerals in these wide contact zones.

Capote Quartzite: The lowest member of the sedimentary series, and the oldest known rock in the district, is the Capote quartzite. It was formerly believed to have been deposited on the Cananea granite, but underground work at Capote mine has shown that the granite intrudes the quartzite. Composition

of the quartzite in the lowest exposed beds suggests that the sediments were derived from a granitic rock, but the granite and pre-Cambrian Pinal schist basement rocks present at Bisbee below the Bolsa quartzite are not known at Cananea. The quartzite is exposed over considerable areas in Capote Basin at surface and in general has a northwest strike and northeast dip. It is well bedded and is now chiefly composed of quartz and sericite with abundant pyrite. The thickness of the formation is obscured by the intrusive granite and by fault contacts, but a stratigraphic thickness of at least 400 ft is exposed northwest of the Capote mine. Valentine[1] believes there may have been as much as 800 ft of quartzite present.

At Bisbee the lowest member of the sedimentary series, the Bolsa quartzite, is given a thickness of 430 ft by Ransome[2] and is assigned to middle Cambrian age. In the Swisshelm Mountains, some 25 miles north of Douglas, Ariz., the basal quartzite member of the Paleozoic series correlated with the Bolsa is about 300 ft thick and in part at least has been intruded by granite at or near the base.

Esperanza Limestone: Immediately above the Capote quartzite, and with no break in sedimentation, there is a series of very thin-bedded, highly altered, and mineralized limestones. Although these beds have been explored underground for about 3000 ft on strike, no trace of original unaltered limestone has been found. The beds are from 1 to 12 in. thick and are completely replaced along bedding by chlorite, epidote, a little garnet, heavy pyrite, magnetite, hematite, some copper and zinc sulphides, and, locally, gypsum. In particular areas the iron content, chiefly pyrite, is very high, and throughout the series there are small isolated pods and lenses of later chalcopyrite with a little bornite and chalcocite. The upper part of the old and very high-grade Capote orebody, mined during the early days of the camp, is believed to have been a secondarily enriched body of relatively high-grade copper ore deposited in the Esperanza beds. The primary ore minerals appear to have been introduced through the Capote breccia pipe which successively cuts quartzite and granite on the Capote lower levels. Ore within the breccia pipe itself on the lower levels was comparatively low-grade.

The thin-bedded highly altered Esperanza beds are more than 300 ft thick and appear conformable with the Capote quartzite. The upper contact grades gradually into thicker-bedded white limestone.

The Abrigo limestone at Bisbee, which succeeds the Bolsa quartzite in the sedimentary column, was given a thickness of 770 ft by Ransome. The series there is composed of very thin-bedded, shaley, flaggy limestones and is assigned to the middle Cambrian. In the Swisshelm Mountains similar limestones above the basal quartzite member are 400 to 600 ft thick.

Crystalline Limestone: Succeeding the Esperanza beds there is a thickness of 300 to 350 ft of gray-white to dark gray crystalline limestones, locally termed *Crystalline* formation. The darker bands are largely in the lower part of the series. Average analyses of these limestones show they now contain up to 12 pct MgO. The Crystalline beds, which can be traced laterally for over 2000 ft underground, extend at surface as an irregular band, interrupted by faulting, from the Veta mine northwesterly across Capote Basin to the Elisa mine. Beds vary from a few inches to about 3 ft in thickness and are conformable with the remainder of the sedimentary series. The strike is northwest, and dips vary from 25° to 45° to the northeast. Generally there has been little mineralizing activity in the Crystalline beds, but some narrow mineralized bands are parallel to bedding, and a few steep mineralized fissures occur. Locally there is considerable alteration with the development of chlorite and epidote near intrusive igneous rocks.

The only fragmentary fossils discovered to date in Cananea have been found in the Crystalline limestones. In the lower beds poorly preserved and very small spirifers and crinoids, which could not be closely identified, resemble Devonian forms. Somewhat better specimens from the upper beds were discovered in 1925 by G. J. Mitchell[3-5] and were identified by G. H. Girty as Mississippian fauna. The fossils included cup corals and a spirifer of the Rockymontanus group.

Sedimentation appears to have been continuous, passing from the underlying thin-bedded Esperanza limestones, tentatively assigned to Cambrian age on the basis of lithology and position in the section, through the Crystalline limestones and into the succeeding beds above. Taking into consideration the few fossils discovered to date, it appears reasonable to place the lower part of the Crystalline series in the Devonian with the upper beds representing a gradational change into the lower Carboniferous. The thickness of the Devonian might be assumed to be from 200 to 250 ft and the upper 50 to 100 ft of the Crystalline beds included with the lower Mississippian.

At Bisbee sedimentation from the underlying Abrigo limestones to the Mississippian Escabrosa appears to have been continuous, but there is no fossil record of deposition of Ordivician or Silurian sediments. The Devonian Martin limestone, between the Abrigo and Escabrosa, has a thickness of 340 ft as determined by Ransome. The contact with the overlying Escabrosa is not clearly marked and is termed gradational. This is also true in the Swisshelm Mountains where about 400 ft of gray limestones can probably be assigned to the Devonian with gradational upper and lower boundaries.

Chivatera Zone: At the upper boundary of the Crystalline limestones an horizon particularly favorable for mineral deposition contains economic amounts of Cu-Pb-Zn sulphides and has been developed in several mines. The zone, called the Chivatera mineral zone, lies between the massive garnetized horizons of the Puertecitos limestones and the underlying Crystalline series. Locally the mineralized bodies extend into the upper Crystalline beds. The zone has a thickness of 50 to 100 ft and the mineralized lenses fade out on strike. The oreshoots roughly follow bedding on dip, and within the mineralized areas the limestone is entirely altered to chlorite and epidote with irregular chalcopyrite, strong sphalerite, pyrite, a little chalcocite and bornite, and a little calcite and quartz. There is some evidence of mineral zoning with an increase in copper minerals at the expense of sphalerite with depth. The zone is not continuously mineralized, but it has been extensively developed in the Veta 5, Chivatera, Hueco, and Campana mines. The position of this horizon favorable to mineral deposition immediately above the upper Crystalline beds, which contain poorly preserved Mississippian fossils, suggests that it also should be placed in the lower Carboniferous.

At Bisbee the lower Carboniferous Escabrosa limestone has proved a very favorable horizon for replacement deposits. There are many important oreshoots extending from the surface exposures of the original Copper Queen orebody on the west, through the Junction mine and other oreshoots localized around the Sacramento Hill porphyry, to the pipe-like Campbell orebody on the east. These have contributed their major copper content from replacements in the east-dipping Escabrosa beds.

Puertecitos Limestone: Above the Crystalline limestones and the Chivatera zone there is a large thickness of conformable, relatively thick-bedded limestones, almost entirely garnetized. Formerly the entire limestone section at Cananea was known as the Puertecitos limestone, the name having been taken from the Puertecitos area where isolated ore lenses in garnetized limestone were mined in the early days. In this paper the name *Puertecitos limestone* will be reserved for the garnetized beds which are the uppermost beds of the sedimentary series.

Bedding appears to be conformable with the rest of the sediments, although the attitude is much less evident than in the Esperanza and Crystalline divisions. The upper limit of the limestones is an old erosion surface upon which large thicknesses of volcanic rocks have been deposited. Extending east and southeast from Capote Basin the most recent volcanic rocks, the Mesa tuffs and agglomerates, cap the garnetized limestones. Within some mineralized areas the volcanic rocks have also been replaced by extensive garnet masses. The garnetized limestones have a minimum thickness of 1000 ft; below areas capped by the Mesa volcanics the thickness may reach 2000 ft.

The age of these upper limestones cannot be closely determined, but the large thickness which has been changed to almost massive andradite garnet suggests that the entire series was chemically similar. Beds of the Carboniferous Escabrosa and Naco limestones in southeastern Arizona do not differ greatly in chemical composition, and as at Cananea the upper limit of the Paleozoic section is an old erosion surface. On this basis it appears reasonable to assign the garnetized Puertecitos limestone to the Carboniferous and to include the Chivatera zone and the upper Crystalline beds in the Carboniferous without further differentiation.

At Bisbee the Mississippian Escabrosa limestone is 600 to 800 ft thick, and the Pennsylvanian Naco limestones, truncated by an old erosion surface, were measured as 1500 to 2000 ft thick. In the Swisshelm Mountains the Escabrosa was estimated at 350 ft and the Naco, only partly exposed, at more than 350 ft.

On the basis of lithologic similarity and the few available fragmentary fossils found in the Crystalline limestones, the Cananea sedimentary section can be tentatively correlated with the Paleozoic section in southeastern Arizona, see Fig. 1. It must be admitted that there are many sources of doubt as to particular boundaries in the conformable series of sediments, but the general comparisons are reasonable. If further investigation makes possible more accurate dating of the Crystalline limestones, the remainder of the sediments can be placed with greater certainty.

The attempt to date the Cananea limestone section has suggested other problems of considerable geologic interest, such as the ages of the volcanic rocks at Cananea and the following periods of igneous intrusions and mineralizing activity.

Surrounding the Cananea Mountains there are wide, gently sloping mesas of unconsolidated conglomerates which reach a thickness of at least 1100 ft at Del Rio about 8 miles north of Cananea. These gravels cover a pediment of the latest Mesa volcanic rocks which lap onto the lower eastern slopes of the Cananea range. The conglomerates are comparable in age to the Gila conglomerate of Arizona.

To the northeast in the San José Mountains, just south of the international boundary, several thousand feet of folded Cretaceous sediments are exposed which correspond to the Cretaceous beds at Bisbee. Toward Cananea, southwest of the San José range, nearly vertical beds of Cretaceous quartzites and shales are found near the Sauz Arroyo crossing on the Naco-Cananea road. These sediments are succeeded to the south and southeast by volcanic tuffs and agglomerates which can be followed to the southeast into the Ajo Mountains about 15 miles east of Cananea. It is probable that the volcanics at the northern end of the Ajo Mountains can be correlated with the Mesa volcanics at Cananea.

In the Ajo Mountains basal quartzites are reported to rest on pre-Cambrian Pinal schist and to be succeeded by Paleozoic limestones above. Cretaceous limestones are also recognized, although generalized relationships have not been established. Northeast of the Ajos, according to N. L. Taliaferro,[6] there is a considerable thickness of upper Cretaceous sediments with some thin, interbedded ash beds.

Some 25 miles south of the Cananea Mountains, at San Antonio Ranch there are extensive surface exposures of shales of probable Cretaceous age; the area between Cananea and San Antonio is largely covered with volcanic rocks.

The problem of the age of the volcanic rocks at Cananea is an interesting one that cannot be fully resolved at present. The Mesozoic sediments found around the Cananea area apparently were deposited through most of the Cretaceous period. Sediments of this age may have been deposited at Cananea, but none are now in evidence. If present originally, they were stripped by erosion before the volcanic rocks appeared. It seems unlikely that the great thickness of Cananea volcanic rocks could have been formed during a period in which there was nearby sedimentation such as appears to have extended through most of the Cretaceous. On this basis the Cananea volcanic rocks are probably not older than late Cretaceous.

As three thick volcanic series have been recognized, the Elenita, Henrietta and Mesa, all separated by erosional gaps, it appears that volcanic activity must have extended over a considerable time. Intrusion of the deep-seated igneous rocks into the volcanics and the following mineralization may have occurred later in geologic history than has previously been believed.

Acknowledgments

The writers wish to acknowledge the assistance obtained from geologic data acquired over the years by members of Cananea Consolidated Copper Co. geologic staffs and consultants. Particular recognition must be accorded Reno H. Sales, V. D. Perry, Wilbur Valentine, and other more recent members of the staff who have contributed to the general knowledge of the district. A. Stoyanow, of the University of Arizona, kindly looked at the few new

fragmentary fossils found. Albert Mendelsohn, President of Cananea Consolidated Copper Co. has kindly given permission for publication of the paper, and only the active support of Mr. Mendelsohn and the company has made possible the continuation of detailed geologic work in the district.

References

[1] W. G. Valentine: Geology of the Cananea Mts., Sonora, Mexico. Geological Society of America Bulletin (1936) **47**, pp. 53-86.

[2] F. L. Ransome: Bisbee Folio, U. S. Geological Survey Reprint (1914).

[3] G. J. Mitchell: Primary Chalcocite at Cananea, Mexico. *Engineering and Mining Journal-Press* (1924) **117**, pp. 880-882.

[4] G. J. Mitchell: Ore Injection at the Cananea-Duluth Mine. *Engineering and Mining Journal-Press* (1925) **119**, pp. 45-48.

[5] G. J. Mitchell: The Geology of Sonora. *Science* (1928) **67**, p. 373.

[6] N. L. Taliaferro: Cretaceous Sediments in Northern Sonora, Mexico. *Journal of Geology* (1933) **41**, pp. 12-37.

W. L. Austin: Ore Deposits of Cananea. *Engineering and Mining Journal* (1903) **76**, p. 310.

R. B. Brinsmade: The Cananea Copper Deposits. *Mines and Minerals* (1907) **27**, pp. 422-424, 465-469.

R. T. Hill: Ore Deposits of Cananea. *Engineering and Mining Journal* (1903) **76**, p. 421.

R. T. Hill: Cananea Re-Visited. *Engineering and Mining Journal* (1903) **76**, p. 1000.

W. H. Weed: Notes on Certain Mines in the States of Chihuahua, Sinaloa, and Sonora, Mexico. *Trans.* AIME (1902) **32**, pp. 428-435.

W. H. Weed: Discussion of article by W. L. Austin, Ore Deposits of Cananea, in *Engineering and Mining Journal*, **76**, p. 310. (See above.)

S. F. Emmons: Cananea Mining District of Sonora, Mexico. *Economic Geology* (1910) **5**, pp. 312-356.

M. J. Elsing: Relation of Outcrops to Ore at Cananea. *Engineering and Mining Journal* (1913) **95**, pp. 357-362.

M. J. Elsing: Secondary Enrichment at Cananea. *Engineering and Mining Journal* (1930) **130**, pp. 285-288.

M. L. Lee: A Geological Study of the Elisa Mine, Sonora, Mexico. *Economic Geology* (1912) **7**, pp. 324-339.

Wm. Catron: Mining Methods, Cananea Consolidated Copper Co., Mexico. U. S. Bur. Mines *Circ. 6247*, 1930.

H. V. Warren: Relation between Silver Content and Tetrahedrite in the Ores of the North Cananea Mining Co., Cananea, Sonora, Mexico. *Economic Geology* (1932) **27**, pp. 737-743.

V. D. Perry: Applied Geology at Cananea, Sonora, in Ore Deposits of the Western States. *Trans.* AIME (1933) **106**, pp. 701-709.

V. D. Perry: Copper Deposits of the Cananea District, Sonora, Mexico. *Copper Resources of the World*, Vol. I, Sixteenth International Geological Congress, 1933.

V. C. Kelley: Paragenesis of the Colorada Copper Sulphides, Cananea, Mexico. *Economic Geology* (1935) **30**, pp. 663-688.

J. B. Tenney: Geology of the Bisbee District, Arizona. *Copper Resources of the World Guide Books.* International Geological Congress, 1933.

Ing. Manuel Santillan: *Copper Resources of the World*, pp. 386-387. Sixteenth International Geological Congress, 1933.

C. U. Cooledge and W. Catron: Controlling Subsidence of a Large Inverted Cone of Barren Rock Lying Above the Orebody, La Colorada Mine, Cananea Consolidated Copper Co. *Trans.* AIME (July 1938) **141**.

W. B. Loring: Geology and Ore Deposits of the Mt. Queen Area, Northern Swisshelm Mts., Arizona. University of Arizona Thesis, 1945.

R. B. Mulchay et al.: Scribner and Chance Mines, Swisshelm Mts., Cochise County, Arizona. Private report, 1949.

Ground Water Control in Underground Mining

by R. C. Mahon

THE importance of ground water control in glacial drift overlying mines is widely recognized. Adequate handling of the problem results in considerable saving in overall pumping costs, as the cost of pumping the larger quantity of water under low heads from surface wells is more than offset by the decrease in the quantity of water to be pumped from the mine under high heads. Eliminating or partly reducing seepage of water through the ledge reduces moisture content of the ore, a factor especially important in iron ore mines, where a reduction of 1 pct in moisture effects a very material saving. An important though indeterminate saving is also brought about by the elimination of water in the working places underground.

The Homer and Wauseca mines of Hanna Coal & Iron Co. at Iron River, Mich., cover 480 acres. When these mines were started it was possible to operate on only one 40-acre tract, as the depth of water over the remainder of the property was too great to permit operation. Although the average depth of the ground water over the area being mined at that time was less than 50 ft it was necessary to pump 1200 gpm from the mine, the greater part from a depth of 750 ft. The slate footwall and graywacke hanging wall are almost impervious, and it was apparent that seepage into the mine was mainly through the iron formation in which the orebodies were located. Analysis of the water in the iron formation showed it to be of the same character as that in the glacial drift, while the water in both walls was heavily mineralized, also indicating that the water passed much more freely through the iron formation.

The first attempt to take off the water above the ledge was by means of drainage drifts driven 15 to 20 ft below the ledge, Fig. 1. Various schemes were used to get the water into these drifts: 1—Holes 15 to 20 ft long were drilled from the back of the drifts to the ledge, but these were difficult to drill and blocked easily. 2—Sections of the drifts were blasted down. This gave a large flow for a time but eventually almost total blockup occurred. 3—Raises 4x4 ft were driven above the drifts to within 8 ft of the

R. C. MAHON, Member AIME, is Superintendent, Homer and Wauseca Mines, M. A. Hanna Co., Iron River, Mich.

Discussion on this paper, TP 3802A, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Jan. 18, 1954. New York Meeting, February 1954.

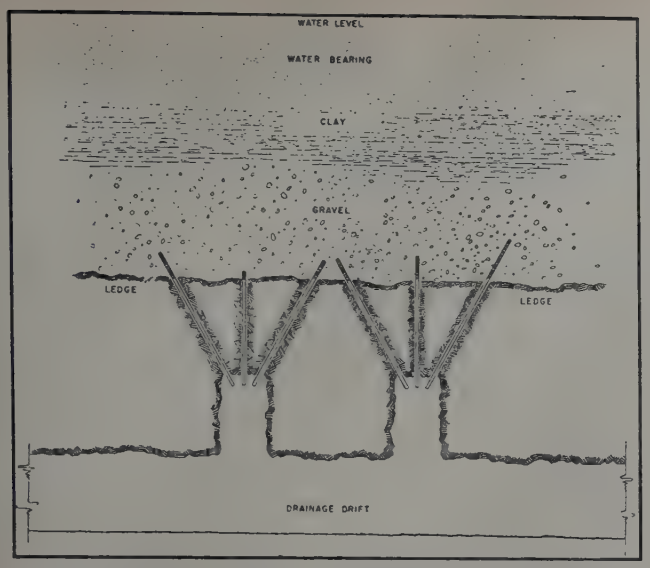


Fig. 1—Theoretical section of the M. A. Hanna Co. Homer mine at Iron River, Mich.

ledge. From these raises holes of 3½-in. diam were drilled to the glacial drift above the ledge and 2-in. pipes perforated at the upper ends were forced up into the glacial drift as far as possible. These holes stayed open for a long time and when blocked could be opened by turning and pounding. By this means 1000 gpm were taken off the ledge. This method did slow the seepage somewhat, but it was evident that it would not solve the problem.

At this time it was determined that a study of the glacial drift was needed, and test holes were put down at strategic points throughout the property, Fig. 2. These holes were drilled with a cable tool Bucyrus Armstrong drill. Eight-inch pipe was used for 100 ft and 6-in. pipe from there to the ledge. In some instances it was necessary to finish the holes using 4-in. pipe. Great care had to be exercised in both the drilling and the sampling. Fine sand formations have a tendency to push up into the casing, and to avoid taking misleading samples it was necessary to slow the pipe and bail it out before advancing. In drilling clay formations the driller had to watch his samples very carefully, as the clay frequently went off with the wash water. All samples were filed for future reference.

These test holes indicated that over a large part of the orebody the water level averaged 150 ft above the ledge. The upper 70 ft was made up mainly of water-bearing sands and gravels. The lower 80 ft was mainly impervious clays with small seams of sand and gravel. From this information it was evident that the water above the clay could not be drained through drifts.

It was then decided to take the ground water off by means of surface wells. The first well was started in June 1929, and by March 1932 five wells were in operation, four of which were sunk only to the clay seam. By now 5000 gpm were being pumped from drainage drifts and surface wells at an average head of 110 ft, and the water pumped from the mine had been reduced from the 1200 gpm in 1929 to 150 gpm. Total pumping costs at that time were 30 pct less than in 1929 because a larger quantity was being pumped from a much lower head. From 1932 to 1936 the mine was idle. Since 1936 eleven wells have been added. Two of the wells sunk to the clay seam have dried up. At present the water level has been lowered to the top of the clay seam, and where the clay is absent average depth of the water above the ledge is now only about 15 ft. The mines are now being operated throughout the 480 acres.

At present 7000 gpm are being pumped from the surface, representing a net gain of about 5500 gpm. Normal rainfall for the district is about 200 gpm per square mile, which would account for a recharge of approximately 800 gpm in the area covered by the mines. Careful measurements have been taken of the rivers and creeks in the area. The normal recharge from the Iron River is approximately 350 gpm, from Sunset Creek 100 gpm, and from the James Mine Creek 100 gpm. During the spring runoff, April to June, this recharge is increased by 500 gpm from the Iron River and 500 gpm from Sunset Creek. There is no evident means of reducing the recharge from the Iron River, but straightening the channels has materially reduced the recharge from both creeks. At present the water level is dropping very slowly, but it is obvious that the drainage gradient is flattening, as a number of wells some dis-

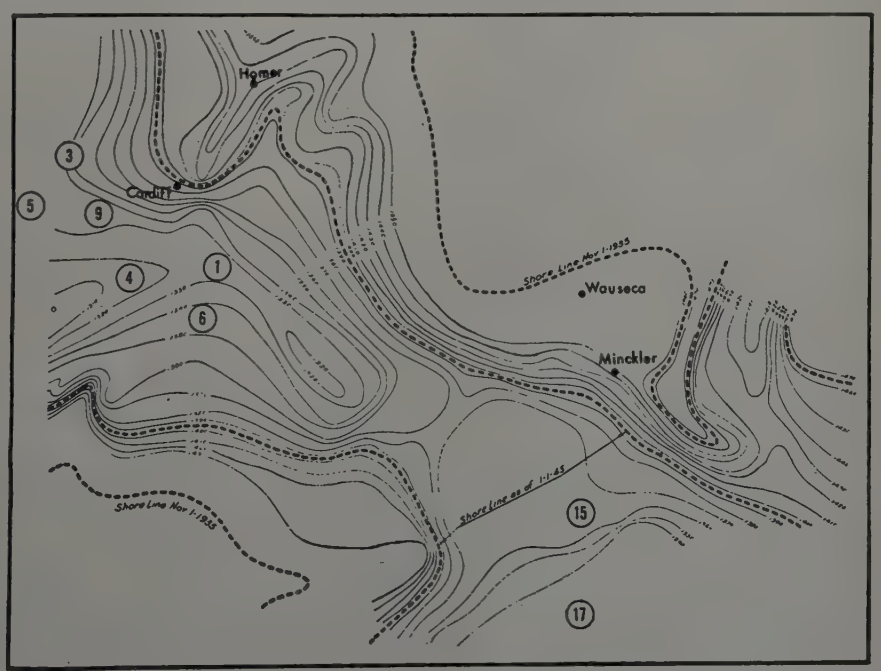


Fig. 2—A ledge contour map of the Homer mine area, Iron River, Mich., showing locations of Homer, Cardiff, Wauseca, and Minckler shafts. Well sites are indicated by circled numbers.

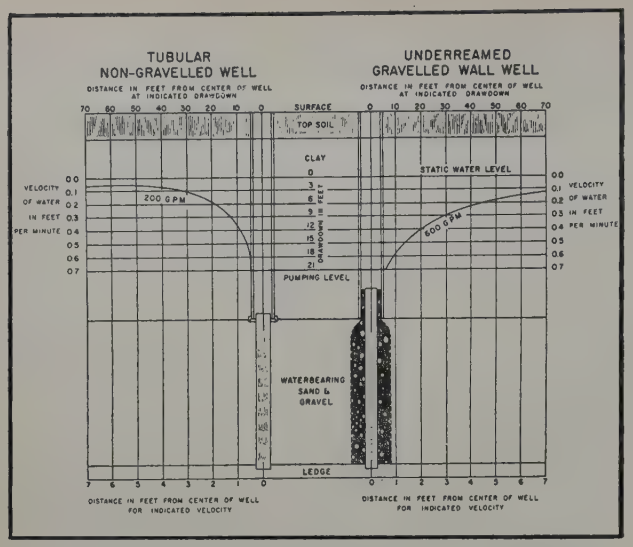

TUBULAR
NON-GRAVELLED WELL
DISTANCE IN FEET FROM CENTER OF WELL AT INDICATED DRAWDOWN
UNDERREAMED
GRAVELLED WALL WELL
DISTANCE IN FEET FROM CENTER OF WELL AT INDICATED DRAWDOWN
SURFACE
TOP SOIL
CLAY
STATIC WATER LEVEL
200 GPM
600 GPM
VELOCITY OF WATER IN FEET PER MINUTE
PUMPING LEVEL
WATERBEARING SAND & GRAVEL
LEDGE
DISTANCE IN FEET FROM CENTER OF WELL FOR INDICATED VELOCITY
DISTANCE IN FEET FROM CENTER OF WELL FOR INDICATED VELOCITY

Fig. 3—Superiority of the under-reamed gravel-wall well over the tubular well is shown here. (Reproduced by permission of Layne and Bowler, Inc.)

tance from the drainage area are drying up. It is expected that when the drainage gradient stabilizes there will be a further drop in the water level throughout the area and a reduction in the amount of water to be pumped. Fig. 2 shows the location of all wells, as well as the drop in the water level in 10-year periods.

Before the wells were sunk it was necessary to determine the best drainage areas, the type and size of well, the location and size of screens, and the method of sinking. Samples from the test areas previously mentioned were examined carefully. In general the best drainage areas were found to be those having formations of gravel and coarse sand without clay contamination.

There has been considerable discussion as to the type and size of well best suited for this kind of drainage. It has been argued that in the long run a large number of relatively cheap tubular wells would be less costly than a smaller number of large gravel-wall wells. The superiority of an under-reamed gravel-wall well over the tubular one is shown graphically in Fig. 3, which represents tests made by the research department of Layne and Bowler, Inc. Although the tubular well is originally less costly it has many disadvantages, particularly the fact that 1—high entrance velocity reduces its capacity, and 2—sand cutting destroys the screen in a comparatively short time and cavities form which eventually cause caving and clogging. The gravel-wall well provides a much greater area around the well from which the water is drawn, thereby lowering entrance velocity and increasing the capacity. Sand cutting and caving are eliminated. This type of well will maintain a greater production over a much longer period of time. In this case it was important that the property be drained as soon as possible and it was decided to use the larger gravel-wall wells.

Well size was governed by depth to ledge, extent of drainage area, and expected inflow.

For the first wells the bailing method was used. A cribbed shaft was sunk to the water level and a casing 30 to 36 in. was placed in the shaft. The casing was heavily loaded with sand bags at the surface and sinking was accomplished by bailing out the material inside the casing. When increasing friction on the casing prevented its sinking further a smaller casing was placed inside it and sunk in a similar manner until the ledge was reached. Clearance was allowed between the casings so that gravel could be fed down outside each one.

Greater capacities were achieved for later wells by use of a rotary drilling rig. The rotary drill may be used initially or a cribbed shaft may be sunk to the water level. A 30-in. casing is placed, and the well is completed to the ledge with the rotary drill. Water is forced through a hollow drill stem, and heavy mud carries the cuttings to the surface. The hole is kept open by the water pressure. When necessary *aqua gel* or its equivalent is added to help keep the water in the hole from seeping out too fast. After the hole has been drilled to the ledge a hydraulic under-reamer is used to increase the diameter in the water-bearing formations. This under-reamer is made up of a number of nozzles directed against the sides of the hole. Water is forced through the nozzles at high pressure and as the under-reamer is revolved the diameter of the hole is increased. After the cuttings have been bailed out of the well the casing, with screens located in the correct places, is lowered into the wall and the space between the casing and the sides of the opening is filled with the proper size of gravel. With this method a much better gravel-wall is obtained and the drainage area of the well is increased.

Recently a new method of sinking wells has been introduced, known as the reverse circulation method. A larger drill pipe is employed, usually 7 in. or more in diam, with a drill-bit on the lower end. A high-capacity suction pump is attached to the top of this pipe, with an impeller designed to take very coarse material. This pump lifts the water to the surface, together with cuttings, which are sized up to about the diameter of the drill pipe. The cuttings are allowed to settle and the water is recirculated. If large boulders are encountered, the drill pipe must be taken out and some other method used to remove them. In sandy formations particularly, or where there are few large boulders, this procedure is much faster than the standard rotary-rig method. With this reverse circulation procedure the casing with its screen and gravel-wall is placed in the same manner as that described for the standard rotary-rig method.

All wells are equipped with vertical centrifugal pumps; these are placed at the bottom of the wells and driven by means of a shaft in a tubing, the shaft being connected to a vertical motor at the surface. In general these pumps have efficiencies of more than 80 pct and operate very satisfactorily. Concrete weir boxes have been constructed at each well and weir measurements are taken weekly. In time the efficiency of the pumps is lowered as the water level goes down and the pumping head increases and the quantity of water pumped decreases. Periodically power readings are taken. When it is found that efficiency has dropped to a critical point the pump is raised and either corrected or replaced with a more suitable unit.

Although advances made in ground water control at the Homer and Wauseca mines have been very satisfactory, further study and experimentation are certainly warranted. Improved methods of sinking the wells to reduce both cost and the length of sinking time are in process of development. Experiments should be undertaken to determine the feasibility and economy of using *aqua gel* or clays to seal the beds of streams and lakes. The problem of recharge is of paramount importance in ground water control.

Properties of Nonmagnetic Taconites Affecting Concentration

by Donald W. Scott and Adam L. Wesner

This paper describes the study of 23 nonmagnetic iron-formation samples from the Mesabi Range and shows the significance of their chemical, mineral, and physical properties in terms of their concentration. Data presented here resulted from a research project carried out from 1942 to 1952 by 10 major producers of iron ore, working in conjunction with Battelle Memorial Institute.

THE inevitable depletion of Mesabi direct-shipping iron ores, hastened by recent wars, has increased the importance of developing methods to concentrate iron-formation materials. Many mining companies, independent research organizations, chemical companies, and equipment manufacturers are devoting attention to this important work.

It is only natural that magnetic iron-formation materials have received major emphasis, since magnetic recovery methods apply and reserves of Mesabi magnetic taconites are large. However, the nonmagnetic* iron-formation materials are likewise believed to represent a large reserve and their study has not been neglected.

* There has been a general tendency to use the terms *oxidized taconites* and *nonmagnetic taconites* synonymously. This is not strictly correct. The term *nonmagnetic taconites* is used herein to mean those formation materials that contain appreciable amounts of the iron in oxide minerals other than magnetite.

Beginning in 1942 a joint investigation was carried out by the major producers of iron ore in the Lake Superior region. In this investigation 23 samples from the Mesabi range, 10 samples from the Gogebic range, 10 samples from the Marquette range, and 5 from the Menominee range were studied. This article describes the 23 nonmagnetic iron-formation samples from the Mesabi range, and shows the significance of their chemical, mineral, and physical properties in terms of their concentration.

Although there is lack of accord on the geological mechanism by which Mesabi *high-grade* ores were formed, it is generally agreed that they were enriched in place and that *taconites* are the parent unenriched or possibly unaltered strata. Mining operations over the years have shown that in addition to the unenriched taconite, which is dense and hard, and the direct-shipping ore, there are transition taconites grading into the friable *wash,* or *intermediate* ores. The transition taconites are less massive than the unaltered taconites and usually coarser-grained. This variation, coupled with the fact that there were no extensive drillings in the unaltered taconite, posed a most difficult problem of obtaining samples that would be representative of large tonnages of the fresh rock. The problem was increased by the tremendous extension of the nonmagnetic formation materials on the Mesabi and by the question of their accessibility to mining. A drilling program was evolved that would surmount these difficulties.

Selection of samples was made by a committee representing producers in the area. The samples were procured from 1—taconite walls near bodies of stripping burden or wash ore and 2—from shafts in the process of being sunk through taconite for drainage or mining operations. Most of the samples were obtained from the cherty horizons because it became obvious early in the program that the slaty horizons represented an exceedingly difficult concentration problem, owing to the character of the gangue minerals and the fine grind required for liberation of the valuable minerals. More samples were procured from the lower cherty horizon than from the upper cherty because it was more accessible to mining.

It is not known how well the samples selected represent the nonmagnetic iron-formation material. However, they were chosen by people well acquainted with the geology of the Mesabi range and the mining problems involved. It is believed that they represent a fair cross-section of the types of materials that will be encountered.

Table I gives data on the origin of the 23 samples and the horizon footages from which they were obtained. Samples 1 through 10, and 15 and 16

D. W. SCOTT and A. L. WESNER, Members AIME, are, respectively, General Manager, Continental Sales and Equipment Co., Hibbing, Minn., and Assistant Chief, Minerals Beneficiation Division, Battelle Memorial Institute, Columbus, Ohio.

Discussion on this paper, TP 3771B, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Nov. 13, 1953. Fall Meeting, Hibbing, Minn., Aug. 31 to Sept. 2, 1953.

(about 1 ton of each) and samples 17 through 19 (20 tons each) were obtained from exposed taconite walls after removal of the obviously weathered material. Samples 11 through 14 (12 tons each) and samples 20 through 23 (25 tons each) were obtained from shaft-sinking operations through previously unexposed taconites. Considering freshness of sample and accessibility to mining, samples 20 through 24 from the Frazer shaft are probably more significant than samples 11 through 14 from the Leonard-Burt shaft, because the former are from the top part of the lower cherty horizon, whereas the latter are from the lower part.

Some of the taconites were a more or less uniform black, red, green, or yellow, and others were banded with light-colored areas of gangue components. Differences in grain size and general structural features were also evident, as shown in Table I. Samples from the slaty horizons were fine-grained and bedding was apparent. Those from the cherty horizons were coarser-grained, although grain size was variable, and more massive, with slight to pronounced banding of alternate gangue and iron minerals in bands up to ¾ in. wide. Samples 8 and 16, in particular sample 8, were coarse-grained and friable, indicative of transition material.

Chemical Composition of Samples

Tables II and III give the respective chemical and magnetic assays. Acid-soluble iron content ranged from 25.3 to 43.4 pct Fe and 17 of the 23 samples assayed 27 to 34 pct Fe. The total iron (acid-insoluble iron also included) was slightly higher in most cases. The ferrous iron content, which occurred in iron silicates, iron carbonates, and magnetite, ranged from 2 to 15.6 pct and seldom exceeded half the total acid-soluble iron content. The ferrous iron content, from 9.9 to 15.6 pct for samples 20 through 24, illustrates the unoxidized character of samples from the Frazer shaft.

The magnetic-iron assays, made on samples ground through 270 mesh, varied from 1.8 to 27.7 pct, accounting for 5 to 85 pct of the total iron. The average for all the samples was 40 pct of the total iron. X-ray and microscopic studies showed that the magnetic concentrates contained some particles that were intimate mixtures of nonmagnetic iron-bearing minerals with enough magnetite to permit recovery in the concentrate by the Davis tube.

The phosphorus content ranged from 0.013 to 0.056 pct. In 17 of the samples the assay was 0.031 pct phosphorus, or less.

Silica, the major gangue constituent in all cases, ranged from 35.1 to 56.1 pct, and from 45 to 52 pct in 18 of the samples. The usual alumina content was of the order of 0.10 to 0.40 pct, exceeding 1 pct for only 5 samples. The carbonate content, representing a major portion of the ignition loss, varied from 0.2 pct to a maximum of 10 pct. The ores with high carbonate content were also high in CaO, MgO, and MnO. The six ores high in CO_2 content were also the highest in Al_2O_3.

Mineral Composition of Samples

Table IV shows the gangue minerals in the taconites and the distribution of iron among the various iron-bearing minerals. Iron-bearing phases included the oxides hematite, magnetite, and limonite, an impure iron carbonate, the mineral minnesotaite, and a limonitic chert. Gangue minerals included quartz, and the silicates greenalite, glauconite, serpentine, stilpnomelane, and clay.

Iron Oxides: Hematite, Magnetite, Limonite, and Goethite; Hematite (Fe_2O_3, 70 pct Fe) and magnetite (Fe_3O_4, 72.4 pct Fe) were usually more abundant than limonite ($Fe_2O_3 \cdot H_2O$, water content variable; 59.8 pct Fe for the formula n = 1.5). The percentage of total iron accounted for in the mineral form of hematite varied from a minimum of 0 to 5 pct for 5 samples to 65 to 80 pct for 7 samples out

Table I. Origin of Mesabi Taconite Samples and Description

Sample	Mine	Horizon*		Horizon, Pct	Description
		Name	Location, Ft		
1	Webb	Upper cherty	Upper 80	46	Fine-grained, massive banded taconite
2	Shenango	Lower slaty	Lower 61	61	Very fine-grained, bedded, slaty taconite
3	Shenango	Lower cherty	Top 35	14	Fine-grained, massive, not banded
4	Shenango	Lower cherty	35 to 122 from top	35	Granular and massive, pronounced banding
5	Adams pit	Lower cherty	60 to 100 from bottom	15	Granular and massive, pronounced banding
6	Mountain Iron shaft	Upper cherty	Bottom 24	16	Fine-grained, bedded, slaty taconite
		Lower slaty	Top 73	44	
7	Mountain Iron shaft	Lower slaty	Bottom 97	59	Fine-grained and laminated
8	Danube	Lower cherty	80 to 100 from top	7	Coarse-grained and somewhat friable banded taconite
9	Corsica	Upper cherty	310 to 350 above quartzite	19	Fine-grained and massive, little banding
10	Mahoning	Lower cherty	135 to 145 above quartzite	4	Fine-grained and massive, little banding
11	Leonard-Burt Drain shaft	Lower cherty	Bottom 45 to 56	4	Very fine-grained, laminated, slaty taconite
12	Drain shaft	Lower cherty	Bottom 32 to 45	5	Very fine-grained, laminated, slaty taconite
13	Drain shaft	Lower cherty	Bottom 21 to 32	4	Very fine-grained, laminated, slaty taconite
14	Drain shaft	Lower cherty	Bottom 9 to 21	5	Fine-grained, massive, slightly banded taconite
15	Adams pit	Lower cherty	75 to 100 from top	9	Fine-grained, massive, slightly banded taconite
16	Spruce pit	Lower cherty	150 to 170 from top	7	Medium-grained, somewhat friable transition taconite
17	Adams-Spruce	Lower cherty	5 to 53 from top	17	Fine granular, massive, banded taconite
18	Adams-Spruce	Lower cherty	51 to 92 from top	15	Fine granular, massive, pronounced banding
19	Adams-Spruce	Lower cherty	92 to 124 from top	11	Fine granular, massive, pronounced banding
20	Fraser shaft	Lower slaty†	450 to 490 below surface	37	Fine granular, massive, moderate banding
		Lower cherty		1	
21	Fraser shaft	Lower cherty	490 to 543 below surface	21	Fine granular, very dense, slight banding
22	Fraser shaft	Lower cherty	543 to 577 below surface	14	Fine granular, massive, moderate banding
23	Fraser shaft	Lower cherty	577 to 640 below surface	25	Fine granular, massive, slight banding

* Precise data on the stratigraphy at the sampling points were not available; that used was of the closest reported point shown on the longitudinal section map by John W. Gruner, Minnesota Geological Survey *Bull. 19.*

† Location tag accompanying sample 20 classified sample as lower cherty; plot indicated that most of sample came from lower slaty. True location questionable.

Table II. Chemical Analysis of Mesabi Taconite Samples

Sample	Total Fe*	Ferrous Fe	Acid-Soluble Fe	Mag. Fe†	Nonmag. Fe†	SiO_2	P	CaO	MgO	Al_2O_3	MnO	Carbonate, CO_2	L.O.I.**
1	32.9	3.0	32.3	11.0	21.3	51.5	0.016	0.01	0.01	0.15	0.24	0.05	1.78
2	25.4	12.8	25.3	5.9	19.4	46.1	0.029	0.34	1.97	2.00	2.18	9.4	9.40
3	27.7	13.1	26.8	5.4	21.4	50.0	0.027	0.01	0.05	0.19	0.74	5.6	7.97
4	33.1	7.4	32.7	19.7	13.0	49.7	0.026	0.03	0.05	0.14	0.52	1.16	1.63
5	34.1	7.6	33.8	17.6	16.2	47.6	0.022	0.02	0.11	0.24	0.49	1.79	1.79
6	32.4	7.7	32.3	18.2	14.1	48.4	0.033	0.15	2.49	0.70	0.18	0.3	0.13
7	32.9	12.8	32.7	27.7	5.0	47.3	0.013	0.03	2.32	0.40	0.14	1.8	0.94
8	36.7	1.8	36.3	1.8	34.5	46.5	0.016	0.22	0.01	0.14	0.08	0.10	0.64
9	44.2	1.8	43.4	8.4	35.0	35.1	0.027	0.08	0.04	0.41	0.10	0.11	1.31
10	34.1	2.8	34.0	12.3	21.7	49.1	0.025	0.23	0.04	0.22	0.04	0.13	1.57
11	30.0	9.4	29.2	16.0	7.1	48.9	0.025	0.34	0.98	1.00	0.72	4.81	5.9
12	28.5	14.4	28.0	11.1	5.4	45.6	0.038	0.55	1.57	1.20	1.16	9.04	9.1
13	27.9	14.7	27.8	5.9	8.8	44.1	0.043	0.50	1.24	1.42	0.97	10.30	11.0
14	28.7	4.8	28.5	9.5	18.5	56.1	0.046	0.01	0.09	1.03	0.08	0.42	1.2
15	31.4	3.6	31.1	4.7	26.0	53.5	0.039	0.01	0.03	0.29	0.11	0.30	1.7
16	36.5	3.2	36.0	3.6	30.4	45.0	0.017	0.01	0.16	0.42	0.52	1.56	2.1
17	34.1	2.9	34.0	11.6	22.4	50.3	0.020	0.01	0.06	0.10	0.21	0.43	1.09
18	33.3	1.5	33.2	6.1	27.1	51.3	0.015	0.01	0.07	0.13	0.06	0.31	1.52
19	32.5	1.9	32.4	12.2	20.2	52.6	0.020	0.01	0.10	0.17	0.12	0.19	1.53
20	33.5	13.3	32.7	25.2	6.5	43.8	0.025	1.04	1.84	0.26	0.80	3.95	3.2
21	28.8	15.6	27.4	17.6	9.8	46.6	0.056	1.07	3.12	0.36	0.44	5.43	4.9
22	29.2	9.9	27.8	18.4	9.4	47.1	0.022	1.52	3.08	0.33	0.37	2.77	2.6
23	26.7	11.9	25.9	11.3	14.6	47.3	0.031	2.17	2.53	0.79	1.17	7.40	7.0

* Determined by fusion. Includes acid-insoluble silicates.
† Magnetic and nonmagnetic assays were determined by Davis-tube tests on 270-mesh head samples. Results reported are calculated as proportional parts of the total acid-soluble iron assays.
** All results for samples dried at 110°C; moisture loss was under 0.5 pct.

of the 23. The distribution of total iron in the mineral form of magnetite varied from as little as 5 to 10 pct for 3 samples to 65 pct or above for 5 samples. There was nothing unusual about the physical and chemical properties of the magnetite and hematite phases occurring in Mesabi taconites, so from a concentration viewpoint the only significance of the distribution described (liberation not considered) is that 5 of the samples, namely, 4, 7, 20, 21, and 22, might be concentrated solely for their magnetite by straight magnetic concentration.

The percentage of the total iron as represented by the mineral limonite ranged from zero for 7 of the samples to a maximum of 20 to 33 pct for 8 of the samples. Generally the hydrated iron oxide was sufficiently crystalline to have the properties of goethite ($Fe_2O_3 \cdot H_2O$ — 62.9 pct Fe), and in this form it might be expected to give no unusual trouble in concentration. In some occurrences, however, the hydrated iron oxide was extremely earthy, probably having coprecipitated with amorphous silica to yield a limonitic chert, the presence of which would be expected to influence the concentration problem.

Minnesotaite:[1] Minnesotaite is a complex iron silicate with a molecular structure similar to talc, except that the major part of the magnesium is replaced by iron. It contains about 28 pct Fe, almost entirely in the ferrous state, 3 to 6 pct MgO, and about 52 pct SiO_2. It is not easy to identify minnesotaite in hand specimens because it resembles earthy chlorite, glauconite, and greenalite, with which it is associated. Of duller greenish-gray color than greenalite, it does not occur in granules but usually is fine-grained and needlelike, with a mottled appearance. Polished surfaces display a bright-green color and a greasy to waxy appearance similar to that of serpentine. It is soft and has 3.0 sp gr.

Under the microscope grains of minnesotaite prove to be acicular, show pronounced anisotropic properties, and appear in sheaflike patterns. When heated to elevated temperatures the material loses its distinct optical properties. The sheaflike pattern persists, but the crystalline structure changes to a semi-translucent, or amorphous-like structure, with a yellow cast almost identical to that of limonitic chert, to be described later.

Minnesotaite accounted for 5 to 15 pct of the total iron in 8 of the 23 samples. On the basis of this distribution and its mineralogy, it is undesirable because the contained iron (30 pct) and silica (50 pct) are chemically combined and cannot be separated from each other by ore-dressing procedures. If the phase is recovered, it will dilute the concentrate with unwanted silica, and if it is sent to waste it will lower the recoverable iron assay in almost direct proportion to the weight percent present, adversely affecting the ratio of concentration and the overall economic picture.

Table III. Magnetic-Tube Tests on Taconite Samples

Sample	Magnetic			Nonmagnetic			Composite	Feed
	Weight, Pct	Assay Fe, Pct	Distribution Fe, Pct	Weight, Pct	Assay Fe, Pct	Distribution Fe, Pct	Fe, Pct	Assay Fe, Pct
1	15.7	67.6	34.2	84.3	24.3	65.8	31.1	32.3
2	12.1	47.6	23.2	87.9	21.7	76.8	24.9	25.3
3	8.2	65.5	20.0	91.8	23.5	80.0	26.9	26.8
4	28.4	69.1	60.3	71.6	18.1	39.7	32.6	32.7
5	25.9	67.5	52.1	74.1	21.7	47.9	33.6	33.8
6	28.8	63.4	56.2	71.2	20.0	43.8	32.5	32.3
7	40.2	67.8	84.6	59.8	8.3	15.4	32.2	32.7
8	2.5	71.8	4.9	97.5	35.4	95.1	36.3	36.3
9	12.4	67.5	19.4	87.6	39.6	80.6	43.1	43.4
10	18.1	67.4	36.3	81.9	26.1	63.7	33.6	34.0
11	26.8	62.9	59.8	73.2	15.4	40.2	28.2	29.2
12	21.2	57.4	43.9	78.8	19.7	56.1	27.7	28.0
13	12.1	53.5	24.8	87.9	22.9	75.2	26.6	27.8
14	21.4	59.1	44.6	78.6	20.0	55.4	28.4	28.5
15	2.9	65.8	6.2	97.1	29.2	93.8	30.3	31.1
16	5.3	66.6	9.8	94.7	33.3	90.2	36.0	36.0
17	16.6	67.7	34.1	83.4	26.0	65.9	32.9	34.1
18	8.8	67.2	18.3	91.2	28.9	81.7	32.3	33.2
19	18.0	66.5	37.7	82.0	24.1	62.3	31.7	32.4
20	36.7	70.4	77.2	63.3	12.0	22.8	33.4	32.7
21	26.2	68.8	64.3	73.8	13.6	35.7	28.0	27.4
22	26.6	70.2	66.1	73.4	13.2	33.9	28.3	27.8
23	16.8	69.1	43.6	83.2	18.0	56.4	26.6	25.9

Tests were made on Davis tube magnetic separator. Samples ground through 270 mesh.

It is questionable, however, whether the behavior of the minnesotaite can be easily controlled. Present indications are that it does not respond selectively either to amine or soap flotation procedures. Heat

Table IV. Mineral Composition of Mesabi Taconite Samples

Sample	Distribution of Iron in Iron-Bearing Minerals, Pct					Gangue Minerals
	Hematite	Limonite	Magnetite	Ferruginous Carbonate*	Iron Silicates†	
1	33	33	33			Essentially quartz.
2	25	20	20	25	10	Clay minerals, greenalite, minnesotaite, and stilpnomelane; little quartz.
3	20	25	20	20	10	Greenalite, quartz, minnesotaite, and stilpnomelane.
4	10	20	65	5		Quartz predominates; minnesotaite and greenalite are of lesser importance.
5	25	15	55	5		Essentially quartz.
6	45		50		5	Clay minerals, greenalite, minnesotaite, and stilpnomelane; quartz is minor.
7	5		85	5	5	Quartz predominates; stilpnomelane, minnesotaite, greenalite, and clay minerals are of lesser importance.
8	85	10	5			Chiefly quartz; minor amount of clay minerals.
9	65	15	20			Chiefly quartz; minor amount of clay minerals.
10	35	30	35			Chiefly quartz.
11	15	10**	55	20		About equal amounts of serpentine and quartz.
12	10	<10**	40	40	< 5	Serpentine more prominent than quartz; minor amounts of greenalite and clay.
13	30		20	50	< 5	Quartz predominates; serpentine also present.
14	65	< 5	30	5		Essentially quartz; serpentine very minor.
15	65	20‡	15			Essentially quartz.
16	<80	5‡	10	5	< 5	Essentially quartz; less than 5 pct serpentine.
17	65	15	20	<2		Essentially all quartz.
18	70	20	10	<2		Essentially all quartz.
19	55	20	25	<1		Essentially all quartz.
20			75	20	5	Quartz predominates; minnesotaite, glauconite, and greenalite also present in minor quantities.
21			65	20	15	Quartz predominates; minnesotaite of lesser importance; glauconite and greenalite in minor quantities.
22	5		65	15	15	Quartz predominates; minnesotaite, greenalite, and glauconite of lesser importance.
23	5		45	35	15	Quartz predominates; minnesotaite, and greenalite of lesser importance; glauconite in minor quantities.

* Iron content of ferruginous carbonate varies from 14 to 35 pct Fe, as compared with 48 pct Fe for pure siderite, $FeCO_3$.
† Includes minnesotaite, stilpnomelane, greenalite, and serpentine.
** A portion in the form of limonitic chert.
‡ In the form of limonitic chert assaying 20 pct Fe and 70 pct SiO_2.

treatment of relatively pure pieces of minnesotaite in air, in a nitrogen atmosphere, or in a hydrogen atmosphere, caused a visual change in color and a change in optical properties but did not make the mineral magnetic. Minnesotaite might respond differently when in association with the other minerals of the ore. This remains to be determined. Although indications are that it will not become magnetic, it might be difficult to keep out of a magnetic concentrate because of its intimate association with other phases that are magnetic, or easily made magnetic. This is discussed under liberation.

Limonitic Chert: Hand specimens of the limonitic chert, of a yellowish-brown color, were in structure similar to flint. Under the microscope the chert appeared to be amorphous earthy limonite. Chemical analyses of specimens, which showed only minor crystalline quartz with the microscope at 450X, assayed 20 pct Fe and 65 to 70 pct SiO_2. The intimate dispersion of either submicrocrystalline or amorphous silica with the iron must account for the excess of silica over the quantity of quartz observed. If the free quartz were deleted, the iron and silica assays would be about the same as for minnesotaite, and because the general appearance of limonitic chert is very similar to that of minnesotaite after heat-treating, what has been termed limonitic chert might originally have been minnesotaite, formed from the alteration of that mineral.

The significant point is that it is not possible to separate the SiO_2 from the Fe_2O_3 in the limonitic chert phase by practicable grinding techniques, and as this reduces the recoverable iron assay the chert is an undesirable iron-bearing phase.

The limonitic chert phase was noted in four samples, namely, 11, 12, 15, and 16. It occurred in quantity only in sample 15, accounting for 20 pct of the total iron, so the iron assay theoretically recoverable in high-grade concentrates is about 25 pct.

Impure Siderite or Ferruginous Carbonate: Pure siderite, $FeCO_3$, contains 48.2 pct Fe, but the siderite occurring in the taconites is relatively impure, containing from as little as 14 pct Fe to a maximum of 35 pct, with varying amounts of MgO, CaO, and MnO. Furthermore, most of the impure siderite in taconites occurs in such intimate association with 10 to 20 pct of submicrocrystalline or amorphous silica that it cannot be observed with the petrographic microscope, much less liberated by grinding.

The impure siderite, or ferruginous carbonate, is soft and does not possess the usual resinous luster or the prominent cleavage normally associated with siderite. Rather, it has a dull grayish-black appearance and a slaty structure similar to slaty ores in general. In the hand specimen, it can be easily mistaken for fine-grained taconites composed only of iron oxides and silica.

The ferruginous carbonate was more abundant in taconite than minnesotaite, accounting for 15 to 50 pct of the total iron for 9 of the 23 samples. These were samples 2, 3, 11, 12, 13, 20, 21, 22, and 23. This phase is undesirable for the same reasons given for minnesotaite but is here of more concern because it is more abundant. Its presence in sample 13 actually reduces the theoretical iron recovery in a high-grade concentrate to about 50 pct, or in other words, it reduces the recoverable-iron assay to about 14 pct.

Just how it will respond in the various concentration schemes remains to be established. Evidence at hand indicates that it is extremely toxic to flotation separations, seriously impairing selectivity and increasing reagent consumption. Excessive reagent consumption is peculiar to this impure siderite, since that consumed by high-grade specimen siderite was only one fourth as much.

Siderite must be decomposed to FeO and CO_2 before the iron can be converted to magnetic form. Decomposition proceeds above 840°F. Below this

temperature siderite is unaltered. Thus with temperature conditions in excess of 840°F, it is to be expected that some of the ferruginous carbonate in ore or mixtures of ores containing ferric oxides and ferruginous carbonates will become magnetic. By control of temperature and other conditions, there is some control over the behavior of the ferruginous carbonate, but as in the case of minnesotaite, its intimate association with other phases which would become magnetic should influence its behavior. Thus while impure siderite does not interfere with reduction-magnetic separation procedures to the extent that it interferes with flotation, it is an undesirable iron-bearing component of taconites.

Greenalite, Glauconite, Serpentine, and Stilpnomelane: Glauconite (a hydrous silicate of iron and potassium), greenalite (a hydrated ferrous silicate), serpentine (a magnesium silicate with some iron replacing the magnesium), and stilpnomelane (a complex mixture of bivalent and trivalent silicates containing some iron) may assay up to 20 pct Fe, depending on the degree of hydration. Because the iron in these minerals is chemically combined with silica, they are all undesirable, but because the iron content is relatively low, they can be classed as gangue minerals. Their presence in the taconites was not serious, because collectively they do not account for more than about 5 pct of the total iron in any sample. This was the case for samples 2, 3, 12, 13, 21, 22, and 23. Reduction procedures did not convert them to the magnetic form.

Quartz: Quartz was the most abundant gangue mineral in the taconites. In only two samples, 11 and 12, was it exceeded in quantity by any other single gangue mineral, namely, serpentine. The quartz occurred mostly in crystals in two size ranges: either coarser than 70 microns (200 mesh) or finer than 10 microns (1600 mesh), with only a small amount between these sizes, but quartz grains commonly occurred adjacent to each other to form large quartz areas. On crushing to 150 or 200 mesh, the finely crystalline quartz broke into smaller particles which were aggregates of finer grains, and these aggregates were found to have a hydrous iron oxide coating on the surface which might decrease selectivity in flotation but would not be important either in magnetic or gravity separations.

Mineral Association

To determine the association of gangue and iron minerals, thin sections were studied as well as sized grains from crushing and grinding tests. Dissemination of iron and gangue phases covered the gamut from extremely fine for samples from the slaty horizons to bands of predominantly iron-bearing minerals ½ to ¾ in. wide in some of the samples from the upper part of the lower cherty horizon. Starting points for the liberation of gangue minerals and iron minerals are listed in Table V. Results of heavy-liquid separations for most of the ores are given in Table VI.

In all the samples, with the exception of those containing iron bands, the major gangue mineral, quartz, was liberated at a coarser size than the iron-bearing minerals. Quartz liberation started at sizes from ¼ in. to 35 mesh, depending on the ore. The float-and-sink separations, made on samples crushed 1/4 in., or to 20 mesh, showed that 1/4 to 1/3 by weight was sufficiently liberated to suggest the theoretical possibility of rejecting primary tailings that might assay 10 pct or less of iron and contain about the same percentage of the total iron. Samples 4, 5, 8, 15, 16, 17, 18, and 19 showed the best liberation of gangue at coarse sizes.

The liberation of the iron-bearing minerals, hematite, magnetite, and crystalline limonite, started at a much finer size than the quartz, except for samples 17, 18, and 19, which contained iron oxide bands. These bands were much less abundant than the highly disseminated areas, but reached a thickness of ½ to ¾ in. They graded into disseminated bands and lean bands. The total amount of the iron values in the high-grade bands was small. Although the float-sink data show that a grind of 100 mesh gave good hematite, magnetite, and limonite liberation from gangue on samples 4, 5, 8, and 9, for most of the samples containing essentially a quartz gangue a grind of 150 mesh would be required. Some iron oxide-quartz ores such as samples 10, 15, and 16 will require a grind finer than 200 mesh.

Table V. Liberation* of Mesabi Taconite Samples

Sample	Iron Minerals	Gangue Minerals
1	Starts at 65 mesh	Starts at 10 mesh
2	Requires grind finer than 600 mesh	Starts at 35 mesh
3	Starts at 35 mesh. Liberation of siderite is poor at 150 mesh	Good at 20 mesh
4	Starts at 35 mesh	Starts at 20 mesh
5	Starts at 35 mesh	Starts at 20 mesh
6	Requires grind 325 mesh or finer	Poor at 100 mesh
7	Requires grind 270 mesh or finer	Poor at 100 mesh
8	Starts at ½ in.	Starts at 20 mesh, good at 65 mesh
9	Starts at 20 mesh	Starts at 20 mesh
10	Starts at 65 mesh	Starts at 35 mesh
11	Starts at 65 mesh. Siderite liberation from iron oxides only 20 pct complete in 150 to 200 mesh size	Starts above 8 mesh
12	Starts at 100 mesh. Siderite liberation from iron oxides only 30 pct complete in 150 to 200 mesh size	Starts at 8 mesh
13	Starts at 100 mesh. Siderite liberation from iron oxides only 20 pct complete in 150 to 200 mesh sizes	Starts above 8 mesh
14	Starts about 65 mesh	Starts about 14 mesh
15	Starts about 65 mesh	Starts about 10 mesh
16	Starts about 100 mesh	Starts at 28 mesh
17	Starts about 8 mesh	Starts above ¼ in.
18	Starts about 6 mesh	Starts above ¼ in.
19	Starts about 8 mesh	Starts above ¼ in.
20	Good magnetite liberation at 150 mesh; only 50 pct liberation of siderite	Grinding to 270 mesh improves slightly the liberation of siderite
21	Good magnetite liberation at 150 mesh; poor siderite liberation	At 270 mesh, 50 pct of the siderite is still locked
22	At 150-mesh grind, almost all the magnetite, about 50 pct of the other iron oxides, and 20 pct of the siderite are liberated	Grinding to 270 mesh improves siderite liberation slightly
23	At 150 mesh-grind, almost all the magnetite, 50 pct of the other iron oxides, and 20 pct of the siderite are liberated	Grinding to 270 mesh improves siderite liberation slightly

* A free particle contains 90 pct, or more, of one constituent.

The textural variation in the taconites which consist principally of iron oxides and quartz is illustrated in the photomicrographs of thin sections shown in Fig. 1. The gray areas and the white areas are quartz and the black areas are iron oxides. The coarse-textured taconite illustrated would result in good liberation if ground to substantially coarser than 150 mesh, the medium-textured taconite would result in good liberation if ground to 150 mesh, and the fine-textured taconite would require a grind substantially finer than 150 mesh for liberation.

For Mesabi Range formation materials containing important amounts of the iron in the form of ferruginous carbonate, or minnesotaite, the grind must be much finer. These phases were liberated from the quartz, serpentine, and other gangue phases at sizes ranging from 10 to 35 mesh, but they were so intimately associated with magnetite and hematite that their liberation in the size range 150 to 200 mesh was only 20 to 30 pct complete. Crushing these

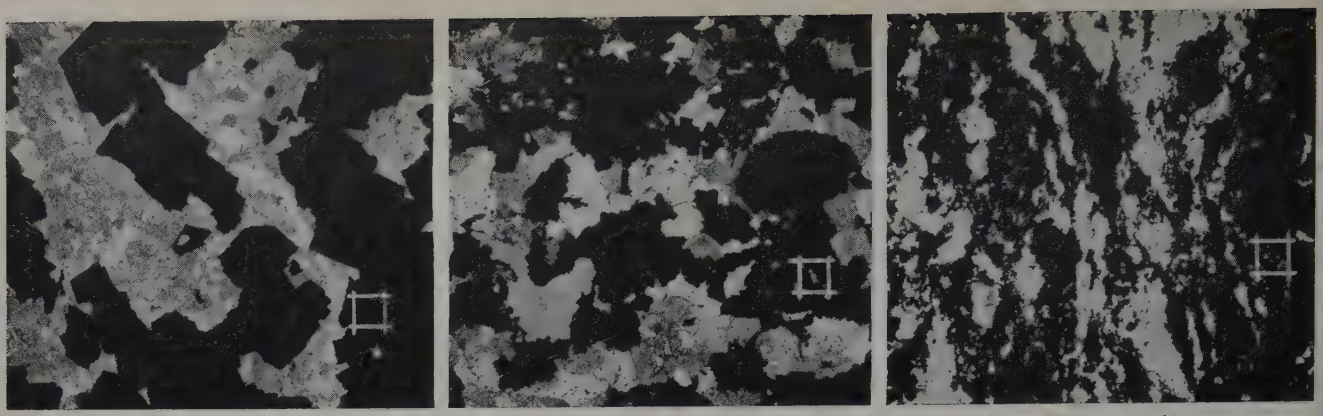


Fig. 1—The above micrographs of thin sections illustrate textural variation in iron oxide-quartz taconites. Gray and white areas are quartz and black areas iron oxide. Left, coarse texture. Center, medium texture. Right, fine texture. Ruled squares indicate a 150-mesh screen aperture.

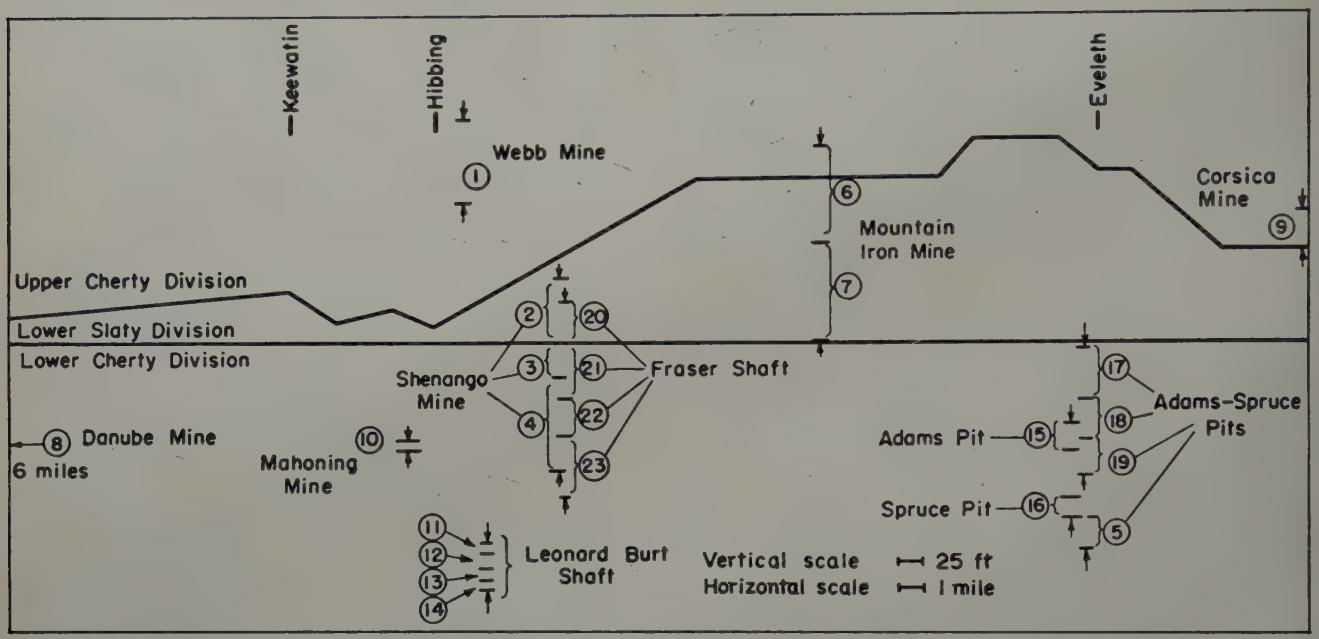


Fig. 2—Approximate stratigraphic and longitudinal location of taconite samples. Horizons located according to longitudinal section map by John W. Gruner.[2]

Table VI. Heavy-Liquid Data on Taconite Samples

Sample	Grind	Product*		Results			
		Sp Gr	Designation	Weight, Pct	Fe, Pct	SiO_2, Pct	Distribution Fe, Pct
14	¼ in.†	Under 2.95	Waste	50.6	14.2		25.3
17	¼ in.	Under 2.85	Waste	23.9	9.4		6.7
18	¼ in.	Under 2.85	Waste	28.0	10.1		8.5
19	¼ in.	Under 2.85	Waste	22.1	10.6		7.1
1	20 mesh	Under 2.95	Waste	25.6	9.9		8.0
3	20 mesh	Under 2.95	Waste	38.5	10.8		15.9
4	20 mesh	Under 2.95	Waste	37.3	6.4		7.6
5	20 mesh	Under 2.95	Waste	34.5	7.6		7.8
8	20 mesh	Under 2.95	Waste	30.9	4.5		3.8
9	20 mesh	Under 2.95	Waste	17.1	8.0		3.2
10	20 mesh	Under 2.95	Waste	20.9	8.6		5.3
11	20 mesh	Under 2.95	Waste	36.5	14.2		18.0
12	20 mesh	Under 2.95	Waste	36.0	12.0		14.5
13	20 mesh	Under 2.95	Waste	24.7	9.4		8.6
14	20 mesh	Under 2.95	Waste	32.5	11.1		12.7
15	20 mesh	Under 2.95	Waste	22.2	9.2		6.7
16	20 mesh	Under 2.95	Waste	16.7	8.6		4.0
1	100 mesh	Over 3.4	Iron-bearing	46.2	57.0	14.9	86.5
3	100 mesh	Over 3.4	Iron-bearing	38.0	47.3	13.9	68.6
4	100 mesh	Over 3.4	Iron-bearing	46.9	60.9	10.1	91.2
5	100 mesh	Over 3.4	Iron-bearing	51.2	60.3	10.5	91.8
8	100 mesh	Over 3.4	Iron-bearing	55.6	63.0	8.3	97.9
9	100 mesh	Over 3.4	Iron-bearing	64.6	61.0	10.3	92.8
10	100 mesh	Over 3.4	Iron-bearing	48.8	57.8	15.6	88.0
17, 18, 19	2 pct +150 mesh	Over 3.0	Iron-bearing	51.9	60.4		95.5

* Only one product is shown in each case, although two or three were made. In the ¼-in. and 20-mesh separations, the low specific gravity or possible waste products are shown, whereas at 100 mesh the results are given on only the heavy or iron-bearing products.
† On sample 14, the —200 mesh fraction was removed.

samples to −270 mesh improved the liberation of the ferruginous carbonate only slightly. Samples containing ferruginous carbonate which fell in this liberation category were 2, 3, 6, 7, 11, 12, 13, 20, 21, 22, and 23.

The samples studied can be classified in three main groups on the basis of mineral composition and physical association: Group A—Ores which are principally a mixture of iron oxides and quartz with good liberation at 150-mesh grinding. Group B—Ores which contain substantial quantities of iron in the form of impure iron carbonate or iron silicates or require a very fine grind for liberation. Group C—Ores falling between A and B in regard to mineral composition or liberation.

Group A includes samples 1, 4, 5, 8, 9, 10, 14, 17, 18, and 19, all from the cherty horizons. Of these, samples 4 and 5 contain a small amount of siderite and/or minnesotaite and approach the intermediate group C ores. The group A ores present the simplest concentration problem because the iron is present in iron oxides which are high in iron content and become liberated with 150-mesh grinding. Concentrate grades of 60 to 65 pct Fe with iron recoveries in excess of 90 pct are theoretically possible.

Group B includes samples 2 and 6 from the lower slaty horizon and samples 3, 11, 12, 13, and 23 from the lower cherty horizon. The first two are included in this classification mainly because they require a grind through 270 mesh, or finer, for mineral liberation. The others not only require a fine grind but also contain appreciable amounts of impure iron carbonates and/or minnesotaite. With the possible exception of samples 2 and 6, theoretically not over 50 to 60 pct of the iron in these samples could be recovered in high-grade concentrates by ore-dressing methods. Obviously a low recovery of iron would increase the tonnage to be mined per ton of concentrate and would affect economics adversely. Present observations indicate that complex ores of the group B type should not be mined if they can be left.

Group C includes sample 7 from the lower slaty horizon and samples 15, 16, 20, 21, and 22 from the lower cherty horizon. Sample 7 requires a fine grind for mineral liberation. Samples 20, 21, and 22 contain impure iron carbonate and therefore their classification in group C is questionable. However, these four samples contain 65 to 70 pct of the total iron in the form of magnetite, which might offset some of the undesirable factors. Ores 15 and 16 contain a significant amount of iron in the form of limonitic chert, from which it cannot be released, and require for liberation a grind finer than is necessary for group A ores and coarser than is necessary for group B. Consequently all the samples in this group are considered borderline. The samples high in magnetite might be amenable to concentration for this mineral only, whereas the others probably could be handled as diluents of group A ores.

Fig 2 shows the approximate stratigraphic and longitudinal location of the various samples. In conjunction with the sample classification presented, it is apparent that taconites vary widely in mineral composition and texture, both areally and stratigraphically, even within a single horizon. Those from the slaty horizon are too finely disseminated to be favorably considered for concentration. However, Fig. 2 also suggests that within a restricted area the variation might be from simple group A to transition group C ores, or from transition to complex group B ores rather than from simple ores to complex ores.

Further sampling and descriptive mineralogical work are required to determine: 1—the extent to which constituent minerals deleterious to beneficiation techniques are present; 2—the relative importance of stratigraphic variation and of oxidation as factors in determining the complexity of the mineral assemblage; and 3—the local reserves, if any, of group A taconites large enough to justify beneficiation plants.

Conclusion

It is possible at present to draw the following conclusions: 1—Any universal method developed for beneficiation of nonmagnetic taconites must be adaptable to a wide variety of minerals and gangue and iron disseminations. 2—Any satisfactory concentrates produced will be essentially iron oxides plus silica, so that a high iron assay will be required to obtain a reasonable silica assay. 3—High recovery will not be possible on ores containing a high proportion of ferruginous chert, siliceous siderite, or minnesotaite.

Gravity concentration might be used for roughing operations. It offers little promise for finishing operations because of low-unit capacity when ores requiring a fine grind are handled. Flotation holds promise for some ores but a change in mineral assemblage can disrupt the separation. To obtain good selectivity, in some instances the grind must be finer than is necessary for mineral severance. Direct magnetic separation, an operable process, is not generally applicable because the magnetite content is too low. Reduction followed by magnetic separation offers several advantages: 1—iron recoveries are expected to be higher in acceptable concentrate grades; 2—a change in mineral composition will not disrupt the process; and 3—the heat-treated taconites are twice as easy to grind as untreated taconites and the liberation is improved about one screen size because of better boundary fracturing.

Reduction studies have been under way to improve thermal efficiency of the conversion of iron-bearing minerals to magnetic form. Of possible application to the problem are the relatively new reduction procedures recently described in the literature: the fluosolids process,[3] the Royster process,[4] and the reduction-oxidation process.[5]

Acknowledgment

The investigation reported herein was a part of a research project on the development of concentrating processes for Mesabi taconites carried on through the period 1942 to 1952. The companies cooperating in the sponsorship were: Armco Steel Corp.; Battelle Memorial Institute; Cleveland-Cliffs Iron Co.; M. A. Hanna Co.; Inland Steel Co.; Jones & Laughlin Steel Corp.; Oglebay, Norton & Co.; Oliver Iron Mining Div., U. S. Steel Corp.; Pickands Mather & Co.; Republic Steel Corp.; and Snyder Mining Co.

References

[1] John W. Gruner: The Composition and Structure of Minnesotaite, a Common Iron Silicate in Iron Formations. *American Mineralogist* (1944) **29**, pp. 363-372.

[2] John W. Gruner: Minnesota Geological Survey *Bull. 19.*

[3] Thomas D. Heath: Process of Reducing Ferric Oxide to Ferrosoferric Oxide. U. S. Patent 2,477,454 (July 26, 1949).

[4] P. H. Royster: Process of Magnetic Roasting. U. S. Patent 2,528,553 (Nov. 7, 1950).

[5] F. M. Stephens, Benny Langston, and A. C. Richardson: Reduction-Oxidation Process for the Treatment of Taconites. Blast Furnace, Coke Oven and Raw Materials Conference (April 20-22, 1953).

Correlation Between Principal Parameters Affecting Mechanical Ball Wear

by R. T. Hukki

This paper presents a series of equations for mechanical ball wear, relating parameters of ball size, mill speed, and mill diameter. The fundamental equation, Eq. 12, presented here is introduced to correlate these basic parameters and thus define and clarify the concept of ball wear. This equation is offered as a general rule, which may be modified to apply to individual problems of grinding.

BALL wear as observed in grinding installations is the combined result of mechanical wear and corrosion. Corrosion should be a linear function of the ball surface available. Ball corrosion, however, has been studied so little that its effect, although of great importance, cannot be included in the analyses given here.

In a separate paper[1] it is shown that

$$n = 0.7663\, n_p \frac{1}{\sqrt{D}} \text{ rpm} \qquad [1]$$

$$P = c_1\, n_p\, D^{2.5} \text{ kw} \qquad [2]$$

$$T = c_2\, (n_p)^m\, D^{(2\,+\,m/2)} \text{ tph} \qquad [3]$$

In these equations

n = actual mill speed, rpm
n_p = calculated percentage critical speed
D = ID of mill in feet
P = power required to operate a mill, kw
T = capacity of a mill, tph
c_1 and c_2 = appropriate constants
m = exponent of numerical value of $1 \leqq m \leqq 1.5$

Exponent m is the slope of a straight line on logarithmic paper relating mill speed (on the abscissa) and mill capacity (on the ordinate).

It is generally accepted, although not sharply defined, that ball wear in mills running at low (cascading) speeds is a function of the ball surface available. Accordingly, the wear of a single ball may be considered to be a homogeneous, linear function of its surface and of the distance traveled. Thus

$$dw = f_1(d^2) \cdot f_2(ds) \qquad [4]$$

where dw is the wear of a single ball in time dt, d the diameter of the average ball in ball charge, and ds the distance traveled by the ball in time dt. Indicating that

$$ds = \pi\, D\, n\, dt,$$

the wear of the average ball in time dt becomes

$$dw = f_1(d^2) \cdot f_2(\pi\, D\, n\, dt)$$
$$= f_1(d^2) \cdot f_2\left(\pi\, D\, c_3\, n_p \frac{1}{\sqrt{D}}\, dt\right)$$
$$= c_4\, d^2\, n_p\, D^{0.5}\, dt$$

The rate of wear of the average ball is given by dw/dt.

$$dw/dt = c_4\, d^2\, n_p\, D^{0.5} \text{ lb per hr} \qquad [5]$$

The weight of the ball charge per unit of mill length is a function of D^2. The number of balls of size d in the ball charge is $= f_3(D^2)/f_4(d^3)$. The rate of wear of the total ball charge equals the number of balls times rate of wear of the average ball. Thus

$$\text{rate of total ball wear} = \frac{f_3(D^2)}{f_4(d^3)} \cdot (dw/dt)$$

$$w_{s\ total} = c_5 \cdot (1/d) \cdot n_p \cdot D^{2.5} \text{ lb per hr} \qquad [6]$$

which is the equation of ball wear in low speed mills.

In a mill running at a low speed, grinding is the result of rubbing action within the ball mass and between the ball mass and mill liners. When the speed of the mill is gradually increased toward the critical, the impacting effect of freely falling balls becomes increasingly prominent in comparison with the rubbing action. Reduction of ore takes place partly by rubbing, partly by impact. The share of the freely falling balls in the reduction of ore reaches its practical maximum at a speed somewhat less than the critical; at that speed grinding by rubbing has decreased to a low value. It may be reasonable to think that size reduction by freely falling balls should reach its theoretical maximum at the critical speed, if the fall of the balls were not hindered by the shell of the mill beyond the top point; grinding by rubbing would cease at the critical speed.

As a first approximation, wear of freely falling balls may be considered to be a homogeneous, linear function of the force at which they strike pieces of rock and other balls at the toe of the ball charge. The force equals mass times acceleration. The mass of a ball is a function of d^3 and its acceleration is a function of the peripheral speed of the mill. The wear of a single ball of size d representing the average ball in a ball charge will therefore be

$$w_f = f_5(F) = f_6(d^3) \cdot f_7(v). \qquad [7]$$

Indicating that $v = \pi\, D\, n$, and $n = c_8\, n_p\, 1/\sqrt{D}$, Eq. 7 becomes

$$w_f = c_9\, d^3\, n_p\, D^{0.5} \text{ lb per hr.} \qquad [8]$$

Total wear of the ball charge equals number of balls times the wear of the average ball. Number of

R. T. HUKKI, Member AIME, is Professor of Mineral Dressing, Finland Institute of Technology, Helsinki, Finland.

Discussion on this paper, TP 3804B, may be sent (2 copies) to AIME before Aug. 31, 1954. Manuscript, Feb. 23, 1954.

balls per unit of mill length equals $f_3(D^2)/f_4(d^3)$. The total ball wear will be

$$w_{f\ total} = c_7\, n_p\, D^{2.5} \text{ lb per hr.} \quad [9]$$

It is seen from Eq. 9 that the total ball wear of freely falling balls is independent of ball size.

It has been stated before that the ball wear of cascading balls is a function of the ball surface (d^2) and that of the freely falling balls is a function of the ball volume (d^3). It is obvious that the exponent of d at speeds between *low* and *critical* must have numerical values between 2 and 3. In the absence of more exact experimental data, the following assumptions are made:

1—At a speed 50 pct of the critical the ball wear is proportional to d^2.

2—At the critical speed, the imaginary action of the balls would be such that the ball wear would be proportional to d^3.

3—At other speeds the ball wear is a power function of d, the numerical value of the exponent q originating from the two given values as shown in Fig. 1.

In Fig. 1 the equation of the line is

$$q = 1 + 0.02\, n_p\,. \quad [10]$$

As can be seen, for $n_p = 50$, $q = 2$ and for $n_p = 100$, $q = 3$. At $n_p < 50$, $q < 2$. An extrapolation of the line gives the value of 1 for q-intercept.

In view of the fact that the line drawn in Fig. 1 is based on certain assumptions, Eq. 10 may also be expressed in the form of the general equation of a straight line as

$$q = c_8 + m'\, n_p, \quad [11]$$

where c_8 is the q-intercept and m' the slope of the line.

Ball wear in low speed (e.g., $n_p = 50$) mills was given in Eq. 6. It may be written as

$$w_{s\ total} = c_5 \frac{d^{\text{II}}}{d^3}\, n_p\, D^{2.5}$$

Wear of freely falling balls was given in Eq. 9. It can be written as

$$w_{f\ total} = c_7 \frac{d^3}{d^3}\, n_p\, D^{2.5}$$

When the exponent of the ball diameter d in the numerator of the above equations is replaced by its value as expressed in Eq. 10, the following relationship is obtained:

$$w_{total} = c_9 \frac{d^{(1\,+\,0.02\,n_p)}}{d^3}\, n_p\, D^{2.5} = c_9\, d^{(0.02\,n_p\,-2)}\, n_p\, D^{2.5} \text{ lb per hr.} \quad [12]$$

Eq. 12 gives the first approximation of mechanical ball wear at any mill speed based on the assumptions indicated above.

If the exponent of d is expressed in terms of Eq. 11, ball wear in its general form will be

$$w_{general} = c_9\, d^{(c_8\,+\,m'n_p\,-\,3)} n_p\, D^{2.5} \text{ lb per hr.} \quad [13]$$

The first approximation of ball wear per unit of ore ground will be obtained by dividing the equation of ball wear (Eq. 12) by the general equation of the tonnage of ore ground (Eq. 3).

$$w_o = \frac{c_9\, d^{(0.02\,n\,-\,2)} n_p\, D^{2.5}}{c_2\, (n_p)^m\, D^{(2\,+\,m/2)}} = c_{10}\, d^{(0.02\,n_p\,-\,2)}\, n_p^{(1\,-\,m)}\, D^{(0.5\,-\,m/2)} \text{ lb per ton.} \quad [14]$$

If $m = 1.0$, w_o will be independent of the mill diameter. In addition, it will not be influenced by the speed term $n_p^{(1\,-\,m)}$. However, owing to the fact that n_p occurs in the exponent of d, ball wear per ton of ore ground increases with increasing mill speed.

If $m > 1.0$, both the speed term $n_p^{(1\,-\,m)}$ and the mill diameter term $D^{(0.5\,-\,m/2)}$ will cause w_o to decrease with increasing values of n_p and of D. On the other hand, the term $d^{(0.02\,n_p\,-\,2)}$ indicates that ball wear per ton increases with increasing speed. From the point of view of ball consumption, it seems probable that in fine grinding it is advantageous to operate at low mill speeds, while the situation may be somewhat different, if not the opposite, in mills grinding coarse ore at high n_p values. In fine grinding ball mills, m = 1.0, but it may have values > 1.0 for coarse grinding ball mills.

According to Eq. 14, ball consumption in pounds per ton increases with decreasing ball diameter, other factors remaining constant. This relationship results from the fact that the effect of ball size on capacity is not considered in the general tonnage equation, Eq. 3. This effect of ball size can hardly be expressed by a simple formula for a general case. However, for ball mills grinding such feeds as rod mill discharge, the grinding capacity seems to be roughly proportional to $1/d^2$. In such a case, the equation of ball wear would be of the form

$$w_o = c_{11}\, d^{0.02\,n_p}\, n_p^{(1\,-\,m)}\, D^{(0.5\,-\,m/2)} \text{ lb per ton,} \quad [15]$$

indicating that reduction of ball size would result in overall reduction of consumption in pounds per ton.

Bond[2] has indicated that an index in terms of kilowatt hours per pound of metal wear furnishes a more useful criterion of ball wear than an index in terms of pounds (of metal) per ton of ore ground. The wear index is obtained by dividing Eq. 2 by Eq. 12.

$$\text{Wear index} = \frac{c_1\, n_p\, D^{2.5}}{c_9\, d^{(0.02\,n_p\,-\,2)}\, n_p\, D^{2.5}} = c_{11}\, d^{(2\,-\,0.02\,n_p)} \text{ kw-hr per lb.} \quad [16]$$

The wear index is independent of the size of the mill. The exponent of d in parenthesis indicates that the wear index decreases with increasing mill speed, in other words, for a certain numerical value of index, less energy is needed if speed is increased.

As a first approximation, liner wear should be proportional to the capacity of the mill, other factors remaining unchanged. Mechanical liner wear and reduction of ore are caused by the same forces acting inside the mill. According to Eq. 3, liner wear may be expressed by a similar formula as

$$w_{liner} = c_{13}\, (n_p)^m\, D^{(2\,+\,m/2)} \text{ lb per hr.} \quad [17]$$

Division of Eq. 17 by the general tonnage equation, Eq. 3, indicates that liner wear per ton of ore ground should be independent of n_p and of D. However, should exponent m in Eq. 17 be = 1.0 for liner wear but > 1.0 for the ore itself (Eq. 3), the equation of liner wear per ton of ore ground would be

$$w_{liner} = c_{14}\, n_p^{(1\,-\,m)}\, D^{(0.5\,-\,m/2)} \text{ lb per ton,} \quad [18]$$

indicating that liner wear would decrease with increasing mill speed and increasing mill diameter. As

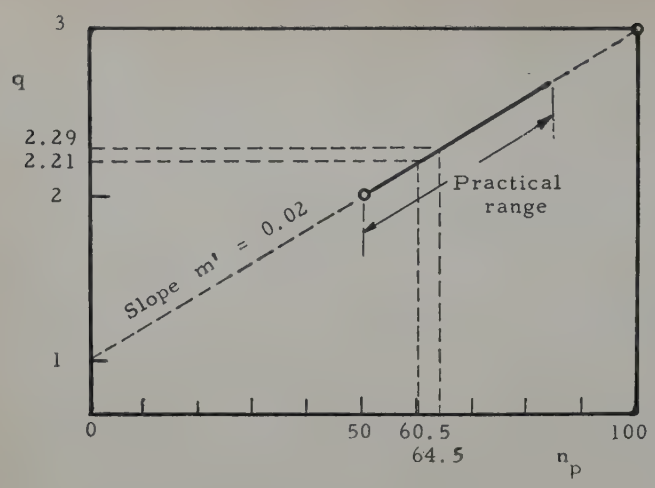


Fig. 1—Assumed relationship between q and n_p.

experimental evidence of liner wear in function of n_p and of D is nonexisting, opinions concerning liner wear cannot be supported by actual test data.

Discussion

The center of interest in investigations concerning ball wear has been the wear as a power function of ball diameter. Numerous studies have been focused on evaluations of the numerical value of exponent q of the ball diameter. Without going more in detail into the respective papers, the outcome of these studies may be summarized as follows: Ball wear proportional to the cube of ball diameter (d^3) has been reported by Davis,[3] and ball wear proportional to the square of ball diameter (d^2) by Ellis,[4] Prentice,[5] Garms and Stevens,[6] Norman and Loeb,[7] and Nordquist and Moeller.[8] The square rule is supported by Taggart[9] for cascading balls; for cataracting balls he gives the exponent as 3.5, probably by mistake. A variable figure such as 2.00, 2.21, and 2.29 is given by Bond[2, 10, 11] with the general remark that $d^{2.00}$ holds at lower and $d^{2.29}$ at higher mill speeds.[10]

As seen, the weight of evidence is strongly in favor of the d^2 rule. This rule, however, must be considered with certain precautions: 1—The actual n_p-value in tests reported is rarely given. 2—The change of n_p with liner wear may not have been appreciated. 3—Corrosion gives an emphasis on the surface rule, but its effect has not been evaluated.

If the evidence cited above is viewed in the light of the analysis presented in this paper, the square rule supports mechanical ball wear in mills running at a speed of about 50 pct of the critical. Exponents 2.21 and 2.29 by Bond would correspond to n_p values of 60.5 and 64.5, respectively, which may be representative of the American ball mill grinding practice. It is probable that in ball mills grinding coarse ore, running at higher speed and using larger balls, the exponent of d would be of the order of 2.2 to 2.5, while in all ball mills used for secondary and tertiary grinding at low speeds and equipped with small balls the square rule would hold in practice, because the experimental difficulties in the verification of the exact value of q for mechanical wear only would be hard to analyze and overcome.

From Fig. 1 it is obvious that at $n_p < 50$, q should be < 2. This possibility may be questionable, because no evidence of the existence of such an exponent seems to be available. On the other hand, whatever information is available, apparently no tests have been conducted at $n_p < 50$.

Investigations concerning rod wear are less extensive. In the controlling rod mill of the Tennessee Copper Co., rod wear is reported by Myers and Lewis[12] to be a function of d^2 (d being in this case the rod diameter) irrespective of mill speed. It would be of major interest to analyze the situation in terms of the parameters included in this paper for coarse-crushing rod mills such as the Sullivan mill.

In the outstanding study of grinding, the Lake Shore Staff[13] found a very useful information by reporting that the ball wear varies closely as the diameter of the mill to 2.6 power. Later, Norman and Loeb[7] observed that the rate at which individual balls wear was faster in mills of large diameter than in mills of small diameter and indicate that theoretically the wear rate of an individual ball will increase as the 0.6 power of the mill diameter. No description was given as to how they came to this theoretical conclusion. As was shown earlier in Eqs. 5 and 6, the relationship between these two concepts should be a simple one. In neither of the papers referred to, however, has the parameter of mill speed been included, although in certain tests at Lake Shore ball wear decreased with decreasing speed. It should be mentioned, too, that liner wear as a function of $D^{2.6}$ was also suggested by the Lake Shore staff.

Conclusion

This short review includes only the most essential information available through extensive investigations made by a number of mill men. It is apparent that the need exists for a basic relationship between mechanical ball wear and the parameters of ball size, mill speed, and mill diameter. The fundamental equation, Eq. 12, is presented here as a general rule to correlate these parameters. Although a basic equation could not be given that would apply in all instances, the rule may be modified for individual problems of grinding.

References

[1] R. T. Hukki: The Significance of Speed of Tumbling Mills on Their Capacity and Power Consumption. Paper to be published, *Trans.* AIME, in 1954.

[2] F. C. Bond: Mathematics of Crushing and Grinding. *Recent Developments in Mineral Dressing.* The Institution of Mining and Metallurgy, London (1953) pp. 101-115.

[3] E. W. Davis: Fine Crushing in Ball Mills. *Trans.* AIME (1919) **61**, p. 250.

[4] O. W. Ellis: Wear Tests on Ferrous Alloys. *Trans.* Institute of British Foundrymen, Thirty-Fourth Annual Conference, June 1937.

[5] T. K. Prentice: Ball Wear in Cylindrical Mills. Abstracted and Reviewed by E. W. Davis. *Trans.* AIME (1946) **169**, pp. 147-154.

[6] W. I. Garms and J. L. Stevens: Ball Wear and Functioning of the Ball Load in a Fine-grinding Ball Mill. *Trans.* AIME (1946) **169**, pp. 133-139.

[7] T. E. Norman and C. M. Loeb, Jr.: Wear Tests on Grinding Balls. *Trans.* AIME (1949) **183**, pp. 330-360.

[8] D. E. Nordquist and J. E. Moeller: Relative Wear Rates of Various Diameter Grinding Balls in Production Mills. *Trans.* AIME (1950) **187**, pp. 712-714.

[9] A. F. Taggart: *Handbook of Mineral Dressing,* pp. 5-27. John Wiley and Sons, New York, 1945.

[10] F. C. Bond: Discussion. *Trans.* AIME (1946) **169**, p. 138.

[11] F. C. Bond: Wear and Size Distribution of Grinding Balls. *Trans.* AIME (1943) **153**, pp. 373-385.

[12] J. F. Myers and F. M. Lewis: Effect of Rod Mill Speed at the Tennessee Copper Company. *Trans.* AIME (1949) **184**, pp. 131-132.

[13] Staff, Lake Shore Mines: Fine Grinding Investigations at Lake Shore. *Trans.* CIM (1940) **43**, pp. 299-434.

Longwall Mining and Mechanization

—— With Special Reference to Nova Scotia

by Frank Doxey

AT Dominion Steel & Coal Corp. it has long been recognized that continued mechanization of mine operations is necessary in the Pictou, Cumberland, and Sidney coal fields of Nova Scotia. The varied physical conditions in these fields call for special consideration of individual cases before planning is finalized. Because standard equipment cannot be procured which would operate successfully, many experiments have been necessary over the years to keep pace with the progress made in other countries.

There are two mines, producing 2000 tpd, located in the Pictou coal field. The field is badly distorted and crossed by many faults. Seams are highly inclined and irregular and vary in thickness from 5 to 40 ft. Entries are difficult to maintain because of squeezing of the coal ribs and movement of the roof and pavement.

Output from the three operating mines in the Cumberland field is 3000 tpd. The field is highly inclined, inclination varying from 12° to 32°. Overlying beds consist of shales and massive sandstone lenses of extreme toughness and are responsible for bumps when the stresses are relieved by extraction. At greatest depth these are among the deepest coal workings in the world. Depth of cover ranges from 2300 to 4000 ft. This prohibits room-and-pillar working and necessitates longwall operation. Working of contiguous seams concurrently to maintain output increases the already difficult conditions.

The Sydney field, with a frontage of about 30 miles, is the most important of the Nova Scotia coal fields. With the exception of one small area it is now wholly submarine. Output is approximately 21,000 tpd. Seams are 2½ to 8 ft thick, and cover in the areas varies from 600 to 2300 ft, with an average of 1500 ft from sea bottom to the top of the seam.

The seams dip in a seaward direction, pitches ranging from 6° to 40°. The shoreline is the last place of entry to the seams and distance from the bank to the working faces is generally over 3½ miles, in some cases as much as 6½ miles. Ventilation is a problem and requires the construction of large permanent airways.

Getting and Loading Coal: In 1925, in view of heavy pressures exerted by thickness of cover overlying the seams, roadways and pillars of the room-and-pillar system being worked began to break up and coal was lost. It was decided that a change in the method of extraction in areas with heavy cover was necessary, and experiments were made with many short walls and longwalls varying between

F. DOXEY is Assistant General Manager, Cape Breton Coal Operations, Dominion Steel and Coal Corp., Ltd., Sydney, Nova Scotia.

Discussion on this paper, TP 3814F, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Dec. 16, 1954. Joint Session CIMM and Coal Div., AIME, New York Meeting, February 1954.

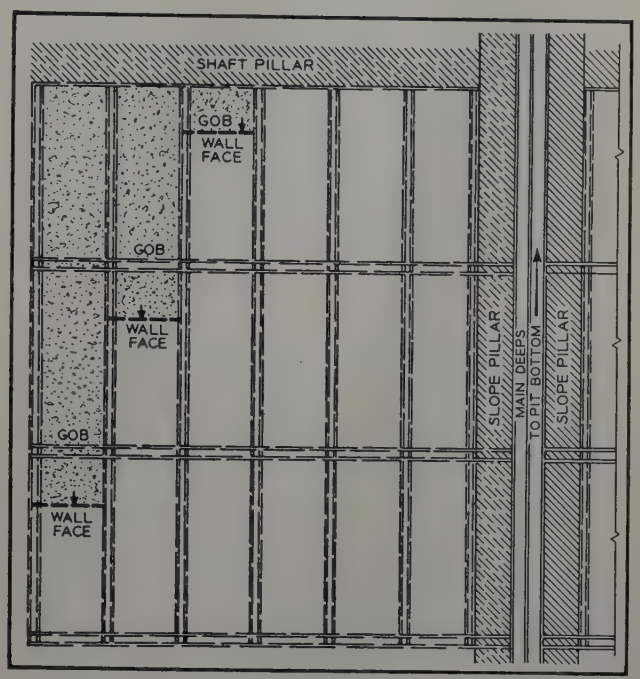


Fig. 1—An example of longwall advance retreating.

90 and 250 ft. Trial and error proved that the best operating length was between 400 and 500 ft, delivering all coal to the dip, with roadways to the face following a level course. The change-over was gradual, and the technique of roof control developed with the system, so that falls to the face are now very infrequent.

An advantage of longwall mining is that it yields 95 pct extraction, especially important in coal seams of high quality or in seams where faults or disturbances restrict the workable areas. This percentage of extraction is based on the fact that the longwall advancing system takes development faces where pitch permits instead of driving headings and leaving roadway pillars. This system yields high tonnage during development and limits loss in extraction to duff left during operations. Accompanying disadvantages, on the other hand, are the heavy construction cost of main roadways and the necessity of driving all new flank face roadways through the gob.

If the main roadways are driven through the solid, and large enough pillars are left on each side for protection against flank face weights, then the width of solid coal is approximately 1700 ft. This represents 10.6 pct of the coal available, or 89.4 pct extraction of the whole. These pillars, however, are of such size that they provide a useful pillar drawing area as a final operation of the mine. It may be that although the seam cannot be generally mined by the room-and-pillar method, it can be adapted to

This page of Mining Transactions AIME follows p. 644. The intervening non-Transactions pages appeared in Mining Engineering.

Fig. 2—Face conditions on a hand-loaded wall. Wall is off line and supports are set haphazardly.

Fig. 3—Face conditions on a mechanized wall. Wall is straight and supports are set to line.

Fig. 4—Chock release for easy withdrawal.

Fig. 5—Back brushing 250 ft from face.

the system during this final operation because of the comparatively short distances through the pillars. It is reasonable to assume that by this method 60 pct extraction of the pillars would be possible, making total extraction of the mine 95.1 pct. Should it be decided to extract pillars by the retreating longwall method, 75 pct extraction of the pillars could easily be obtained, making a total extraction of 96.6 pct. Allowing 5 pct for duff losses in working, extractions would be 90 pct and 91 pct respectively.

Advancing System: The advancing system, operated in most longwall mines, is determined by the following factors: 1—desirability of production in quantity as soon as workings are clear of shaft or slope pillars; 2—inability to hold headings in the solid, either during drivage or face extraction; 3—high initial cost of preparing a mine for retreating; 4—liability of seam to spontaneous combustion.

Most mines except Cumberland in the Nova Scotia areas practice longwall advancing. Walls are between 400 and 500 ft long and work singly or in multiples of these lengths, 2500 ft being the longest length advanced each day.

Longwall Retreating: Apart from the high initial cost, longwall retreating appears to have advantages over longwall advancing, provided that development headways can be driven in series and fully mechanized and that restricted output during the development period is not a serious factor. There are, however, many problems accompanying the system, such as ventilation in gassy seams, material supplies, heaving pavement, the necessity of running long conveyor belts from faces to auxiliary haulage levels, or the difficulty of holding loader ends in the pillars outside the working face. It has been found from experience in Nova Scotia that longwall retreating has no operating advantage over the advancing system, except where the physical conditions are ideal, or in some special application such as that in the Cumberland field where the workings are subject to bumps.

In this case, when longwall advancing was worked the whole of the working face was affected, sometimes very seriously, when a bump occurred. In view of this it was decided to try the retreating system; after the change it was found that when a bump took place the roadways leading to the face were the places damaged and the working face was scarcely affected. Removal of the bump area from the working face to places more remote from the workmen was a safety step which fully justified the system.

Longwall Advance-Retreating: Where development headings are easily driven and maintained, this method can be a useful compromise between the two systems, panel development being prepared

near the shaft or slope pillars. Development headings proceed to the lateral boundary or boundaries, opening up panels of faces on one or both sides, the faces being retreated to the levels and back to the main haulageway while further development is proceeding inby for replacement purposes. Where total extraction is essential, faces can be extracted as shown in Fig. 1.

Choice of Face Line: Generally speaking, the best line of face is at right angles to the cleat for machines similar to the Meco-Moore, Gloucester getter, and Dosco miner, while for machines of the German plow and Samson stripper type the line is apparently better parallel to the cleat.

In flat seams either system is reasonably easy to plan, but in steep work such as obtains in the Nova Scotia fields the line of face is usually determined by the dip rather than by the cleat, as disadvantages of working heavy machines across outweigh advantages of working with the cleat or across it.

Roof Control: The roof of the longwall workings in Nova Scotia mines is controlled by the building of chocks and packs in the gob and the setting of timber at the face.

Built of old stone and wood, the packs are 12 ft wide and in most cases are put on at 50-ft centers. The chocks, built at 7-ft centers, are 6x6-in. hardwood, 2½ ft long in the Sydney field and 4 ft in the Acadia and Cumberland fields. Chock releases are placed at convenient height for withdrawal.

Timber at the face is set at 4-ft intervals. When a large area of roof is exposed where the men are working, it is essential that as near perfect control as possible should be kept. If this is achieved the getting of the coal, of course, is made easier.

It is necessary, therefore, to maintain the fracture line of the roof behind the chocks in the gob. This can only be achieved if the chocks are well built and a high standard of chocking, packing, and timbering is maintained.

The chocks are advanced daily after the packs have been extended and the roof stone allowed to fall behind the chocks. The timbers also are drawn off in the gob. It is very important that all back timber and chocks be drawn systematically to keep the weight evenly distributed and equally important that all pack wall sides be well built and in a straight line.

This enables the roof to break off along the side walls of the packs in addition to breaking off behind the chocks, thus forming a series of corrugations at right angles to the face which give support to the upper measures.

Fig. 6—View of roof-bolted back brushing 250 ft from face. Side bolts and a section of the Sydney Mines gate belt loader are also shown.

When the system of packing for any longwall section has been determined, packing plans should be made and marked up weekly, showing pack positions and advance, and any other condition which has occurred during the week, such as falls to face. These plans compile a history which can be very useful in assessing the reason for any given face condition at any time.

The position of the chocks in the waste, with the resultant fracture line, determines to a very great degree the actual roof condition at the face and the load and position of the roof over the coal being worked. This is very important where mechanized mining is practiced and where roof stresses are released rapidly. Figs. 2 and 3 show clearly the effect of roof control on face conditions.

Many systems of timbering, packing, and chocking have been or are being practiced in longwall mines. Some of these are outlined as follows: 1—packing and chocking, with wooden face supports. 2—packing and chocking, with steel face supports. 3—chocking and steel face supports where the caving system is used.

These systems are practiced in varying forms in Europe and Britain, No. 3 being the least used. All have been tried in Nova Scotia mines.

Fig. 7—Side breaking away over roadside packs.

Fig. 8—Roadside after side-bolting with 8-ft bolts.

Fig. 9—Roof bolting in roadway 800 ft from working face. This is not a substitute for packing and chocking on roadsides.

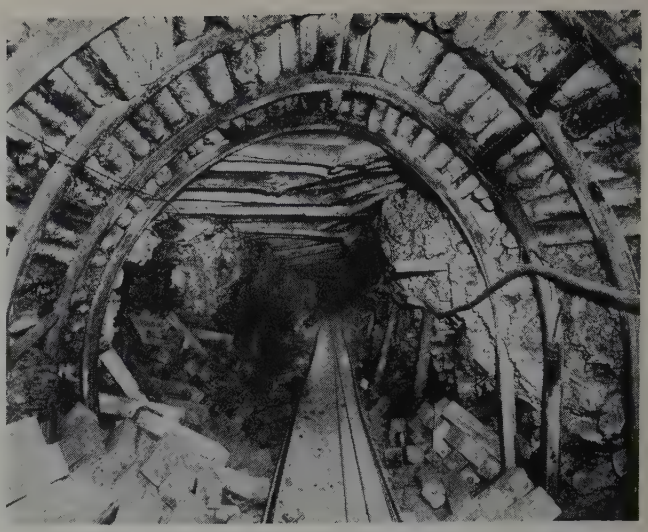


Fig. 10—A roadway where bolts have not been used, 800 ft from the working face.

Rigid and yielding props play a very important part in roof control, and many fine examples are in existence. The hydraulic prop has become very popular in Britain, and many hundreds of thousands of rigid and mechanical yielding props are in use in that country. With the hydraulic prop, initial installation cost is very high, maintenance is of vital importance for efficiency, and any losses in withdrawal are very costly. In spite of this, however, reduced cost per ton can be achieved over a period of time, provided losses are kept to a minimum.

It must be borne in mind that with thick seams the question of handling heavy steel props must be considered, as well as safety of the props in steeply pitching seams. System No. 1, that is, packing, chocking, and wooden timbering, is the one now used in Nova Scotia, owing to seam thickness and pitch, hard and slippery pavement, and face falls when the caving system was tried. Over the years the system has worked reasonably well and has improved considerably since the introduction of chock releases, Fig. 4.

At one period roof bolting on longwall was tried; this was not satisfactory as a substitute for conventional support, but short bolts offered distinct advantages where soft shale overlying the seam had a tendency to fall with the coal. Roof bolting is also desirable across roadheads where moving conveyor equipment to new positions requires the removal of props. No matter what system of face and gob support is used, it must have the effect of creating a fulcrum behind the working area on the face and so relieve the pressure as to eliminate roof breaks at the coal face.

Pneumatic Stowing: Pneumatic stowing of longwall workings has been practiced for several years in Europe and Britain. It has been used with particular success in the Doncaster and South Wales coal fields in Britain, in the Doncaster field as a spontaneous heating deterrent and in South Wales as a means of extracting a seam full of breaks which was sandwiched between a roof and pavement full of breaks caused by working of contiguous seams.

In the Doncaster field, where the Barnsley seam is highly spontaneous, strip packing and roadway packing is done by pneumatic means. At Bullcroft colliery a completely self-contained system is in operation. Mine waste is loaded into cars and taken to an underground crushing plant. After crushing it is returned in cars to the stowing machines located in the face roadways, tippled, and stored. The waste used is —2½ in. This system, while expensive to establish, has reduced the incidence of spontaneous heating, decreased the number of men in the packing team on face, dispensed with a large crew of men whose sole duty was to deal with heating, and provided continuity of output.

At the other collieries in the district, and also at the South Wales mines, waste is provided for stowing from the surface spoil heaps.

Roof Control in Roadways: As mentioned previously, the future condition of roadways is chiefly determined by roof control at face. When the roof is exposed it should be supported immediately and packs put on as soon as possible to control subsidence and prevent bed separation. The rib should be protected by a pack to take care of any cutoff along this side. This is eminently desirable for all roadways but is to be emphasized for steep measures. To prevent side pressure and ease roadway maintenance the amount of brushing taken down at the face should be kept to an absolute minimum consistent with the area required for ventilation and transportation.

Roadside packs should be well built and solid throughout to give maximum support to the roadway, break off the wastes on each side, and prevent leakage of air through the gob. This last factor is particularly important in seams liable to spontaneous combustion.

The roadway between the first back brushing and the face end should be given maximum support that is reasonably easy to withdraw. This can be done in many ways, for example: 1—straight wooden booms pocketed in the side of the roadway and middle set where necessary; 2—straight or cambered steel I-beams pocketed in the side; 3—wood or steel herring-bone supports; 4—steel arch girders set on stilts or loose rock, soft wood, and wire bags filled with waste.

Back brushing supports are of a more permanent nature and may take the form of steel arches; straight or cambered girders, propped or pocketed in the side; steel and wood *moll* supports; or roof bolting. Wood and stone packs on roadsides, straight wood booms supporting first brushing, and steel arches or roof bolting at first back brushing are the methods generally used in Nova Scotia. Roadside

packs also have an important part in roof control at the face and maintenance of a good roadway behind.

Generally speaking, the first back brushing in gob roads is about 250 ft from face where the seam is 5 to 6 ft thick and could be a longer or shorter distance as the seam thins or thickens, Fig. 5. Where a number of faces are advanced together, care should be taken to keep them as nearly in line as possible. Should steps develop between faces, the roadway will be subject to the weight of the leading face and will be further disturbed by the trailing face, instead of being allowed to settle uniformly. This condition invariably produces bad roadways.

In seams 5 to 6 ft thick it is usually necessary to take one or more further back brushings, particularly in roadways in the center of a multi-wall section in comparatively flat seams and in the center and to the dip of the center roadway in steep measures.

Roadway Roof Bolting: Where proper roof control is exercised, so that breaking off takes place in the gobs alongside the roadway and the roof spanning the roadway is left unbroken, roof bolting at the back brushings can be used to distinct advantage, Fig. 6. The effect of subsidence is not felt nearly so badly in a roadway supported in this manner as it is in a conventional steel arched roadway. Spare roads or bypasses are much more readily maintained and traveling between cars much easier. Bolts must be long enough to maintain a compact body. Eight-foot bolts are used in the Nova Scotia applications.

It is common for the sides of longwall roadways to break away back over the packs for a distance of 3 to 5 ft, Fig. 7. This condition can be prevented by the insertion of side bolts, Fig. 8, a comparatively new system of support for longwall roadways that shows great promise.

When it is considered that four bolts and a wooden boom replace one arch and lagging, it will be seen that the saving in material cost is tremendous. Cost of roof-bolted sections is 33 pct cost of arching.

Great care must be exercised in choice of roadways for roof bolting, Figs. 9 and 10, and it must be emphasized that roof bolting cannot act as a substitute for good packing and chocking on road sides.

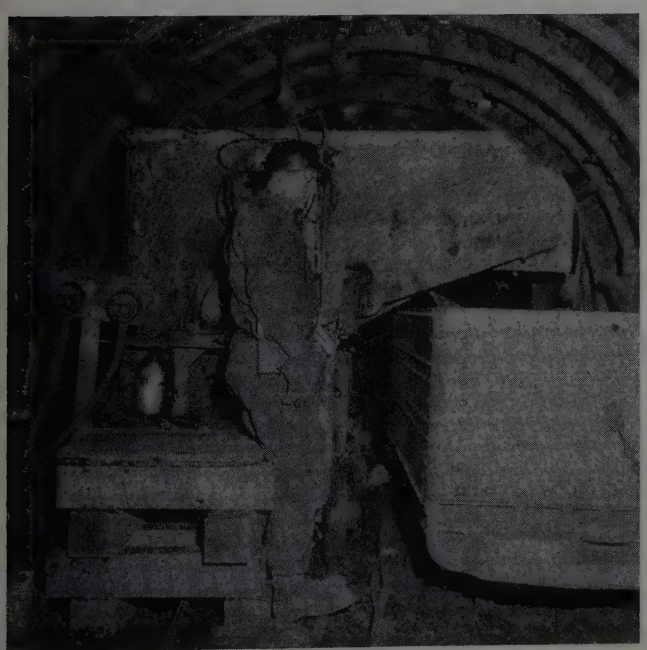


Fig. 11—Discharge end of the Sydney Mines loader.

In gassy seams pillars left between one face or set of faces and the next prevent gobs from settling evenly and cause roof breaks which may convey methane from any bed separation or from nearby overlying seams into the adjacent roadway. If pillars must be left in a gassy seam to prevent fire or flooding or to provide roadway support, methane drainage is desirable, depending on the quantity to be piped to surface or into the return a safe distance from the face. In Nova Scotia mines there are two such installations for piping gas into the returns. The installations are new and sufficient detailed information regarding drainage is not yet available, but evidence is that the system will be invaluable.

Roadway and Face Transportation

When longwall mining was first introduced in Nova Scotia, coal-cutting machines were used to undercut the coal, which was then drilled and brought down by explosives to be handfilled onto shaker conveyors.

At first the coal was loaded from the shaker conveyors directly into mine cars. This system worked reasonably well where the faces were short but fell far below the speed and efficiency necessary for longer faces and greater output. In view of this, gate belt conveyors were introduced which picked up the coal from the face conveyors and transferred it to a suitable loading point outby.

The conveyor was extended each day following a strip off the face and the loader end remained in its semi-permanent position, to be moved only when

Fig. 12—Main haulage diesel installation.

Fig. 13—Auxiliary haulage diesel installation.

conveyor length, varying from 1200 to 2000 ft, resulted in overload. Such loader ends easily handled large outputs, but in steep thick seams, where heavy maintenance is necessary, the length of the conveyor soon became a distinct handicap, hindering repair work and damaging conveyor structure. The company therefore developed a fixed length gate conveyor, Fig. 11, known as the Sydney Mines loader because of its place of origin.

The gearhead of this conveyor is especially constructed to transfer the coal at right angles to side load into mine cars. Because the side-loading chain conveyor is clutch-driven from the main belt conveyor gearing, cars can be uniformly loaded and spillage during spotting is prevented, the bin being of sufficient capacity to hold the coal while cars are being changed. A further clutch-driven rope drum is used for self-hauling the conveyor, which is on wheels and track-mounted, in or out as required.

A tail end auxiliary scraper chain conveyor is driven through sprocket and chain by the tail drum of the belt conveyor, placing coal on the belt conveyor in the direction of travel. This reduces belt wear and also keeps the receiving end of the gate conveyor at a very low level, which in turn assists materially both in the discharge of coal from the face to the gate conveyor and in the loading of coal from the *Dosco* miner onto the face conveyor.

This fixed-length conveyor, usually 300 ft, moves ahead each strip off the face; the back brushing, being just ahead of the loader end, must be done each shift to allow the conveyor to move in. This accomplishes the back brushing and systematically maintains the same distance between back brushing and face. Further, the load end operator works under permanently supported roadway with plenty of room for handling mine cars. Where the conventional gate belt system is used a rigid schedule of back brushing and repairs should be maintained.

Auxiliary Haulage: In the early days horses were used for hauling cars along loading levels, later being replaced by endless or main and tail haulage. During the last four years Diesel locomotives have been introduced into the Nova Scotia mines to haul between the loader ends and the main deep landings and have proved immeasurably superior to the rope systems previously used.

Introduction of the diesel locomotive has brought in its train more meticulous tracklaying, fewer derailments, and fewer haulage accidents. In longwall working, where the roads run through the gob, diesels are infinitely safer than electric trolley wire locomotives and far more flexible. Objections are sometimes raised about the safety of this type of transport, but from experience it has been found that the safety of the diesel locomotive leaves nothing to question if good roads, good track, good maintenance, and good ventilation are insisted upon by operators and inspectors.

Table I. Comparative Statistics for Diesel and Rope Haulage

Diesel Haulage			Rope Haulage		
Accidents	Days Lost	Tons Per Accident	Accidents	Days Lost	Tons Per Accident
2	28	135,974	8	468	28,289

Table I, above, shows typical diesel and rope installation accident statistics in the same mine in Nova Scotia. Figs. 12 and 13 illustrate two diesel installations.

Because of the reduced number of roadways in the longwall system of mining, special care should be taken that plans provide for main roadway sizes adequate to carry all ventilation necessary for the life of the mine, cutting down air pressure loss between pit bottom and working face to a minimum. High water gages should be avoided wherever possible, particularly in seams liable to spontaneous heating. There should be enough face splits to provide adequate quantities of air with low velocity.

In pitching seams, however, where ascensional ventilation is necessary, some compromise may be necessary on these points. Where mechanical mining is taking place, low velocities should be aimed at to assist in dust suppression measures. Degassing is a move in this direction also.

The room-and-pillar system in comparatively flat seams with good physical conditions is easy to mechanize, but the longwall system does not lend itself so readily, particularly in steep work. Over the past few years this matter has been given careful consideration in longwall countries and various machines have been devised and manufactured.

The coal plow which met with reasonable success in the soft, uncleated coals in Germany was not so successful in the harder cleated bituminous coals in Britain. Of all the British and European machines, the British-manufactured Meco-Moore longwall cutter loader apparently has been the most successful in British mines but has been adaptable only in certain conditions and in the flatter seams. The coals of Nova Scotia, while classed as soft coals, are very similar to those of Great Britain, being fairly hard with well-defined cleavage.

The steeper gradients of the seams, however, posed a problem which required special consideration, since no suitable machines were available. In view of this, Dominion Steel & Coal Corp. engineers designed the machine known as the Dosco miner, based on the Joy Manufacturing Co. principle of passing a number of cutting chains in a shearing action through the coal face.

In 1949 a prototype machine was manufactured by Trenton Industries Ltd., a subsidiary of Dominion Steel & Coal Corp., and put to work in an open strip mine to test its qualities. After getting and loading some 20,000 tons of coal the machine was completely redesigned and the present machine shown in Figs. 14, 15, and 16 was evolved. Eleven such machines are now working and giving very reasonable results, the best daily output to date from one machine being 587 tons in an 8-hr shift and the best weekly output 2860 tons in five shifts.

The machine consists of a main body split in two parts, one part sliding on the other. The whole is mounted on a main frame and caterpillar-propelled. The cutting head is made up of seven cutting chains of the Austin Hoy ball-joint type which carry 536 carboloy-tipped cutting bits.

The machine receives its power through two trailing cables extending from the magnetic switch gear situated in the loader gate to a switch on the machine. The cables, No. 2/0 and No. 6 B & S, are identical in construction apart from the power wire size, both having six wires: three individually screened power wires, two pilot wires, and one ground wire. Trailing cables are 75 ft long to facilitate handling. The pilot wires in the cable form part of an intrinsically safe operating circuit. The operator, by

Fig. 14—A view of the Dosco miner, designed by engineers of the Dominion Steel & Coal Corp.

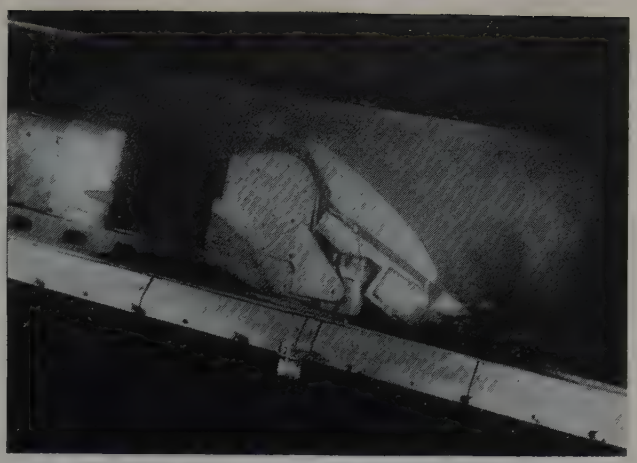


Fig. 15—The Dosco miner cutting head sumped 18 in. into coal. Note water spray for dust suppression.

Fig. 16—The Dosco miner cutting head at top of lift. Eleven of these machines are now in use.

Fig. 17—Magnetic switches for use with Dosco miner are operated by snap action switches on the miner.

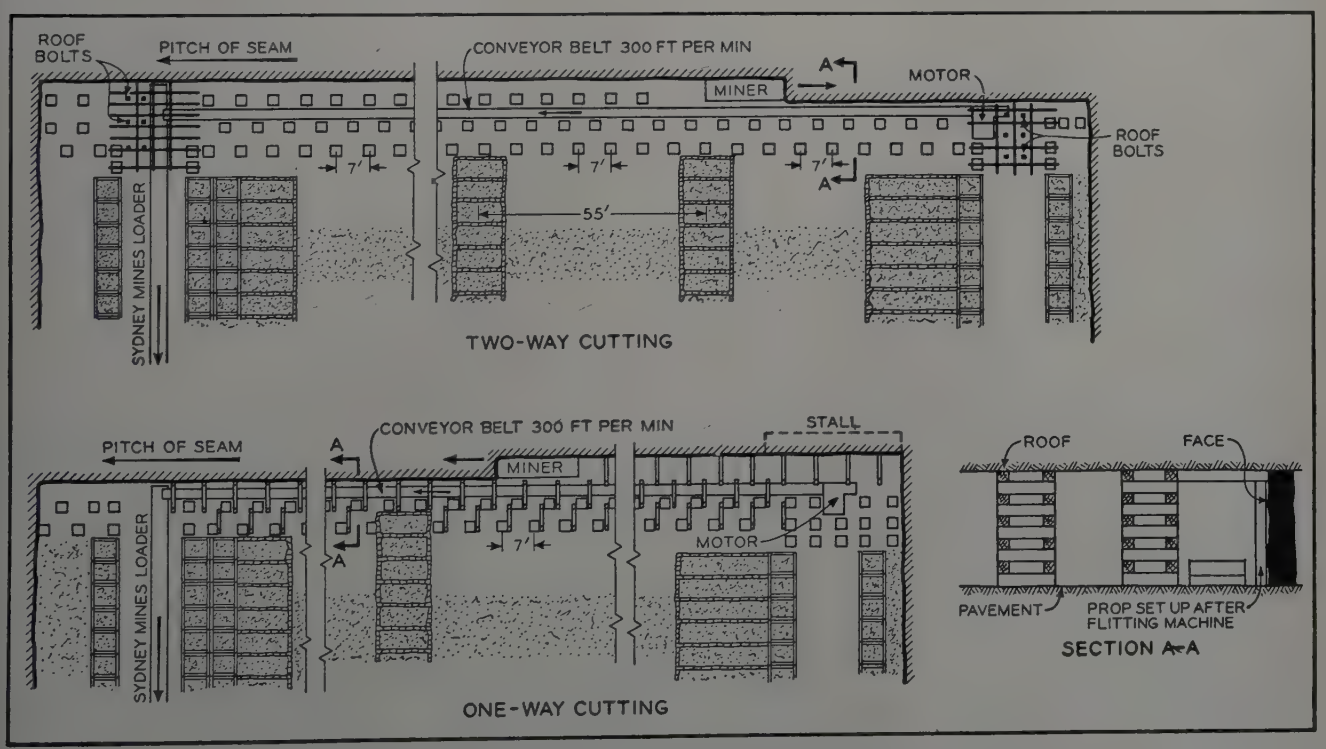


Fig. 18—The one-way and two-way systems of the Dosco miner longwall operation.

closing or opening snap action switches, makes or breaks the pilot circuits which control operation of the switches outby the loader end, see Fig. 17.

The cutting chains carry the coal back to a built-in variable-speed (0 to 500 fpm) hydraulically driven conveyor in the machine, which in turn discharges it to the face conveyor.

The 2/0 cable supplies the power to two 75-hp water-cooled motors, each coupled through cone worm gear cases to the main driving shaft in the cutting head. The No. 6 cable supplies the power to a 30-hp water-cooled motor for the driving of three hydraulic pumps. Two 24-gal pumps supply power to the sumping cylinders, jib-lifting cylinders, rear jacks, and caterpillar crawlers, and one 11-gal pump supplies power to the cross conveyor. After passing through the motors the water is atomized through sprays to control dust made during cutting.

The 75-ft lengths of cable attached to the machine are changed over to fresh couplings as the machine proceeds along the face. Seventy-five feet of cutting with an average of a little over 1 ton per ft yields approximately 80 tons of coal. The size of trip servicing the loading end is therefore made to fit within this tonnage limit, so that cables are changed over while trips are being changed, thereby avoiding loss of time during cable changing. The cutting cycle of operation is as follows:

1—Move whole of machine to the face on caterpillars with cutting head in down position.

2—Sump cutting head into coal a distance of 18 in. This is done hydraulically, the cutting head and conveyor sliding forward on guides. Telescopic splined shafts allow continuous drive while this is taking place.

3—Lift cutting head through the coal to top of seam, or to predetermined height.

4—Retract cutting head.

5—Lower cutting head to pavement.

6—Move ahead on caterpillars and repeat.

The whole cycle takes 25 to 30 sec to complete, so that cutting speeds up to 3 fpm are easily obtained. In a 6-ft seam this means a production of about 4 tons per min, but since less than half the time for the cycle is productive the rate of production is 8 to 10 tons per min.

The shaker face conveyor, which was adapted to the machine, was completely unsatisfactory and had to be replaced by a 30-in. belt conveyor with a running speed of 350 fpm.

Too much stress cannot be placed on the importance of attention to detail in setting up the belt conveyor, which should be built to a line each time it is moved. It should be level across and as free from undulations as practicable.

Two systems of loading are in operation, one-way and two-way loading. In the one-way system, which is used in the steeper mines, the machine starts cutting from a stable at the top of the face, the timberers systematically setting supports to the mine regulations. In this system one end of the boom is hitched into the face, the gob side end being supported by a prop 5 ft from face. This allows for flitting back the machine to the top of the wall at completion of the cut. The stable in this case is cut by machine, drilled and loaded by three men. It is 30 ft long so the machine can be maneuvered into its new position. In the two-way system the machine cuts its own stable. The support method in these systems is shown in Fig. 18.

The face conveyor is driven from the tail end, that is, from the top end of the face, to allow the belt to be kept near the pavement throughout the whole length of the face and to facilitate loading at the mine loader receiving end. The nine-man machine crew for the one-way system includes 2 operators, 3 timbermen, 3 stablemen, and 1 official. The six-man crew for the two-way system consists of 2 operators, 3 timbermen, and 1 official. These crews replace 50 men: 40 loaders, 2 cutters, 4 drillers, and 4 shotfirers.

It will be seen that on the mechanized faces there is a substantial saving in loading manpower. Table II shows a typical crew setup in each system. These figures show the mechanized face forces to be only 57 pct of the hand-loading face forces.

Table II. Crews for Mechanized Loading and Hand-Loading System

Crewmen	Mechanized Loading	Hand Loading
Overmen	2	2
Shotfirers	2	6
Conveyor operators	2	1
Electricians	1	
Timberers	3	
Operators	4	
Maintenance men	1	
Pipemen	2	1
Belt mechanic	1	2
Material men	3	4
Panmovers	10	13
Chock drawers and packers	18	18
Stall	3	
Brushers	3	3
Cutters		2
Helpers		4
Loaders		40
Total	55	96

Shifts are unproductive chiefly in the brushing, packing and chocking, and moving conveyor categories, and it is here that the next steps must be made to reduce force. One man in each roadway could be saved at the front brushing by the use of mechanical equipment for dirt disposal. The number of packers and chockers may be further reduced by the development of mechanical or pneumatic machinery for pack-building and use of mechanically or hydraulically operated chocks and props.

As mentioned previously, the shaker conveyor could not deal with the loads from the Dosco miner and was replaced by a belt conveyor. The labor required in both cases is heavy and a much more flexible and easily removable conveyor is desirable. The German panzer armored chain conveyor is becoming more and more popular in longwall workings in Britain and many revised versions are being manufactured by British mining machinery companies to meet the prevailing conditions.

These developments are being carefully watched, for it is with the conveyor of robust construction and adequate capacity that can be moved bodily by hydraulic, pneumatic, or mechanical means that further reductions can be made in oncost labor. One manufacturer has promised to attempt to develop a conveyor of this type, of capacity sufficient for the Dosco miner operation. It seems feasible, therefore, that cost results usually associated with the room-and-pillar system may be obtained in deep mining by longwall methods where the room-and-pillar system is not practicable, provided that every operation is as fully mechanized as possible. Mechanization of the ancillary operations requires further thought and development, and it is the policy to continue with experiments to bring this mechanization about.

Rock Bolting in Metal Mines of the Northwest

by Lloyd Pollish and Robert N. Breckenridge

SUCCESS in any underground mining operation is determined by accessibility of the orebody, which in turn is dependent upon maintenance of passageways to the mining zones and temporary support of the voids caused by extraction of ore. This is accomplished by one or a combination of the following methods: timbering, back-filling, pillaring, or, more recently, rock bolting.

Timbering has usually been the principal means of maintaining these underground openings necessary for mining operations. Timber, however, does not prevent ground movement beyond the scope of localized sloughing, which is indicated by the gradual failing of the timber itself. Besides this, timbering has always been a costly process, and with the decline of available supplies of timber close to the mining areas, mining men have constantly sought other methods of controlling ground.

Rock bolting is now replacing timbering at an ever increasing rate. Experience has proved that this form of ground support is just as applicable to blocky igneous rock as to stratified rock. Besides preventing sloughing of the walls and back of underground openings, Fig. 1, rock bolting has a stabilizing effect on the surrounding ground in much the same manner that steel reinforcing rods add to the strength of concrete structures. Further, rock bolting is flexible and may be applied to any shaped excavation, whereas timber sets are in a fixed pattern and the ground must often be changed to conform with this pattern.

Rock-bolting installations were made in metal mines of the Northwest as early as 1939. An exhaust air crosscut was driven that year in one of the Butte mines of the Anaconda Copper Mining Co. The crosscut was rock-bolted and gunited at the time it was driven and is still being used to exhaust hot humid air from the 3400 level of the Belmont mine. It is interesting to note that no sloughing or caving has taken place in the 14 years it has been open. Even though these early installations of rock bolts were successful, few men recognized their potentiality until recent years, when the coal mines started their programs of mechanization and the great trend toward roof bolting began.

In some areas of the Northwest stopes that previously required heavy timbering and close backfilling are now being mined by the more economical cut-and-fill and shrinkage methods. When used in conjunction with timbering, rock bolting increases the efficiency of the operation by decreasing hanging wall dilution and by making it possible to blast larger rounds.

Most of the rock bolts installed to date in mines of the Northwest have been the 1-in. diam slot and wedge type, but there has been a recent trend toward using the ¾-in. diam expansion shell bolt shown in Fig. 2. In addition to these commercially manufactured steel bolts, wooden bolts have been used with considerable success by the Day Mines of Wallace, Idaho.

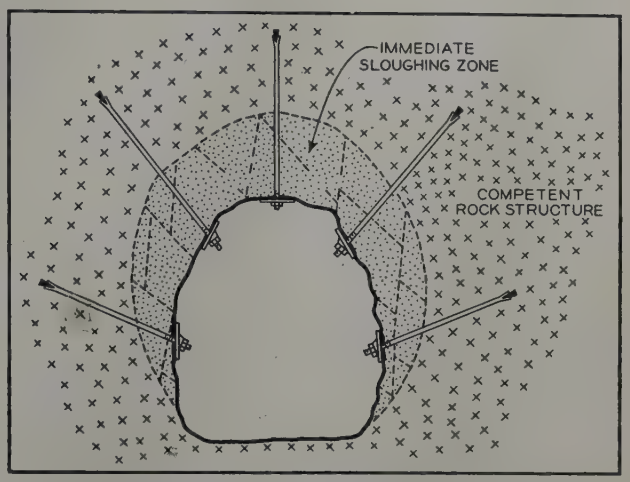


Fig. 1—Rock bolting prevents sloughing in underground openings and stabilizes the surrounding area.

Installation of the slot and wedge type requires three distinct operations, with tools for each operation: 1—drilling the hole to proper diameter and depth, 2—setting the bolt, and 3—tightening the nut. Holes are drilled and bolts set with pneumatic rock drills. A number of setting or driving tools have been used successfully, but most follow the same general pattern. Usually the driving tool is designed to accommodate a short length of drill steel on one end and the rock bolt on the other end. In this manner the hammering effect of the rock drill is transmitted through the steel and driving tool to the bolt. When machines not having stop rotation are used, slippage is allowed between the driving tool and bolt or between the drill steel and driving tool. The rock bolt nuts are tightened either with pneumatic impact wrenches or with hand wrenches. Impact wrenches are desirable because they are faster and assure adequate tightness.

Expansion shell bolts have the following advantages over slot and wedge rock bolts: 1—No special equipment other than a wrench is needed for their installation. 2—Installation is faster. 3—They are removable. 4—Holes need not be drilled to a specific depth as the expansion shell will anchor anywhere along the length of the hole. These advantages are offset somewhat by the lesser strength of the bolt, since expansion shell bolts are generally made from ¾-in. diam steel as compared to 1-in. diam steel for the slot and wedge type. One manufacturer, however, is now fabricating expansion shell rock bolts from steel of high tensile strength, which gives this ¾-in. bolt a much greater strength than that of the mild steel bolt.

Table I illustrates tests made by the Anaconda Copper Mining Co. to determine the proper hole size to use with various types of bolts and to determine

L. POLLISH, Associate Member AIME, is Assistant Research Engineer, Engineering Research Dept., Anaconda Copper Mining Co., Butte, Mont. R. N. BRECKENRIDGE, Member AIME, is Mining Engineer and Shaft Foreman, Sunshine Mining Co., Kellogg, Idaho.

Discussion on this paper, TP 3813A, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Feb. 3, 1954. New York Meeting, February 1954.

Table I. Pull Test Data on Expansion Shell and Slot and Wedge Rock Bolts

Bolt No.	Hole Diam, In.	¾-In. Expansion Shell Bolt				1-In. Slot and Wedge Bolt			
		Type A		Type B		Type C		Type D	
		Tons Pull	Result	Tons Pull	Result	Tons Pull	Result	Tons Pull	Result
1	1¼	19	Broke		Hole too small	20	Broke	21	Slipped
2	1¼	19	Slipped		Hole too small	21	Broke	22	Slipped
3	1⅜	17	Broke	14	Broke	18	Broke	15	Slipped
4	1⅜					20	Broke	17	Slipped
5	1 7/16	10	Slipped	13	Broke	15	Slipped		
6	1 7/16					18	Broke		
7	1½	5.5	Slipped	12.5	Slipped	18	Broke		

their points of failure. Bolts were anchored in the usual manner and tension was applied by means of a hydraulic roof bolt puller until failure occurred. Results show that the average failure for all ¾-in. expansion shell bolts tested occurred at 13.8 tons, and for 1-in. slot and wedge bolts at 18.6 tons.

Treated wooden bolts, employing the slot and wedge type of anchorage and constructed from straight-grained fir, have been used successfully in faulted areas and in *short ground*. Their main application seems to be in wet ground where the ability of the wood to absorb moisture causes them to expand, affording contact with the sides of the hole along the entire length of the bolt. Pull tests, made in hard dry ground, resulted in slippage of the bolt when the applied stress was only a small percentage of the calculated tensile strength of the wood.

Rock bolting increases efficiency of stoping operations. In square-set stoping it reduces hanging wall dilution, eliminates the need for heavy timber, and prevents movement of false hanging walls which cause timber to ride and break. In cut-and-fill stoping it reduces hanging wall dilution, eliminates the need for temporary safety stulls which often impede scraping operations, and makes possible the breaking of larger rounds. In shrinkage stoping it facilitates rapid mining of an orebody, increases productivity per manshift, affords a low unit cost of mining, and effects a saving in the transportation of materials.

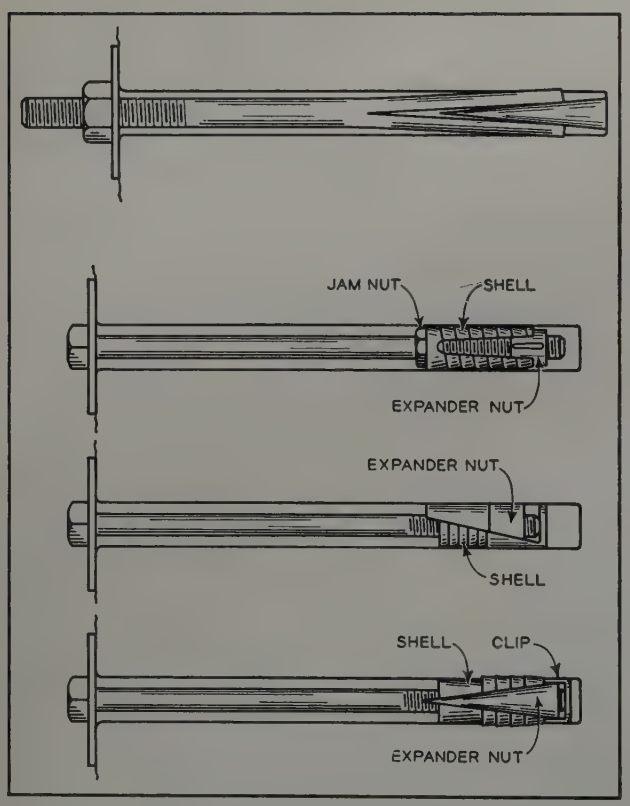


Fig. 2—An example of the slot and wedge type of bolt is shown above and three expansion shell types below.

Substitution of rock bolts for timber in development headings and haulageways reduces the required excavation by approximately 30 pct without intruding into the necessary opening, see Fig. 3. Installation of timber in waste headings requires that this extra rock be broken to accommodate timber sets, so the purpose of rock bolting is to make possible the excavation of a minimum size cross-section and secure the perimeter, or sloughing zone, by binding it to the naturally self-supporting rock structure surrounding it. Rock bolts support the ground at each point of installation, whereas support given by timber is confined to the blocking, which is normally at the junctions of the cap and posts. Further, ground may be bolted within a few feet of the breast with no adverse effects from blasting the succeeding round.

Rock bolting practices at the Butte operations of the Anaconda Copper Mining Co. and applications of rock bolting at the Sunshine Mining Co., Kellogg, Idaho, were taken as typical examples of practices in metal mines of the Northwest. The following accounts show how rock bolting has been employed effectively with various types of stoping operations as well as with almost every other type of underground excavation, see Table II.

Table II. Distribution of Rock Bolts for a Two-Year Period, Anaconda Copper Mining Co.

Place Used	Total Used, Pct	
	1953	1952
Sills	70.7	80.9
Stopes	24.8	5.9
Raises	1.7	1.7
Pockets	1.6	5.4
Shafts	0.3	3.5
Miscellaneous	0.9	2.6

The Butte mines have used 73,645 rock bolts since the start of the present program in late 1950 and are now using about 5000 bolts per month for all types of underground excavations. Both the 1-in. diam slot and wedge type and the ¾-in. diam expansion shell type are used. Standard lengths are 3, 4½, 6, and 8 ft. Although most bolts are still used for sill development work, an increasing number are being used for ground support in stopes, replacing square-set timbering. This is due in part to Anaconda's present program of mining narrow veins by the more economical horizontal breast cut and hydraulic fill method, using rock bolts for temporary support of the hanging wall and back.

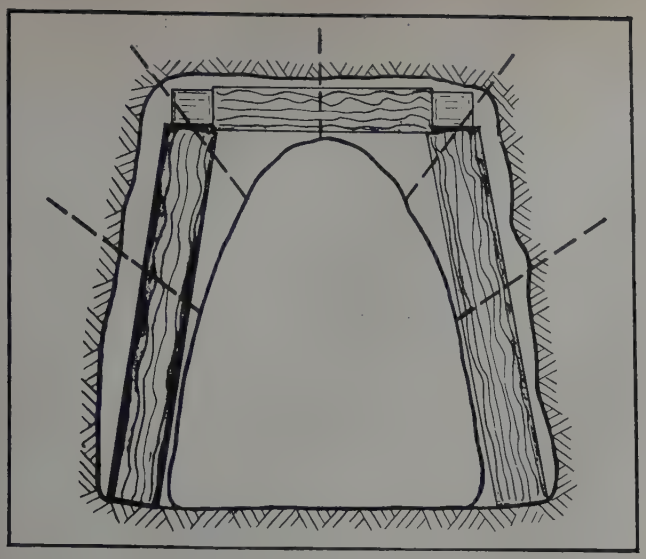


Fig. 3—A 9x7-ft rock-bolted cross-section superimposed on an 11x9-ft timbered cross-section.

Rock Bolting in Stopes: Fig. 4 illustrates the rock bolting procedure used at the Mt. Con mine in stoping by cut-and-fill method. When necessary the hanging wall and back are bolted immediately following a blast to give support during the subsequent scraping, filling, and drilling operations. Bolts are 6 ft long and are installed on 5-ft centers horizontally and 4-ft centers vertically. Push feed drills are used exclusively in these stopes for drilling and rock bolting.

Bolts are installed by regular contract miners and both the slot and wedge and expansion shell types have been used successfully. The expansion shell bolt is ideal in this application, since the ground remains open only long enough for the cut to be made. They may be removed from the back before the blast is made to keep them from falling into the muck pile where they would impede scraping.

Rock bolting has also been applied to square-set stopes with considerable success. When it was noted that the timber in several stopes at the Mt. Con mine had begun to ride and break, 6-ft rock bolts were driven into the hanging wall. This practice was continued for each successive floor. At the third floor apparently all ground movement had ceased. This indicated that a fracture or clay seam, parallel to the plane of the vein, had caused a false hanging wall which was gradually slipping downward as the supporting ore was removed, a common occurrence in Butte ground. In these cases the shear strength as well as the tensile strength of the steel rock bolts was being utilized in keeping this false hanging wall in place.

Rock Bolting Large Excavations Prior to Concreting: The condition of the country rock found in the Butte mines usually necessitates timber support for any large underground excavation. Fig. 5 illustrates a method used to excavate an underground hoist room on the 4000 level of the Mt. Con mine. The excavation was enlarged from two 9x7-ft crosscuts 50 ft long with connecting crosscuts at both ends. The ground was first supported with timber, but when this began to fail, 8-ft rock bolts were driven into the walls and back and the timber used only for staging as the excavation progressed. The room was then entirely concrete-lined. The protruding ends of the bolts reinforce the arched roof slab and were useful in hanging the reinforcing rods. The absence of timber blocking against the ground simplified the placing of the concrete forms.

The Kelley mine 600 level ore pockets are concrete-lined, rail-reinforced, cylindrical excavations with 14-ft diam and 65-ft depth. The pockets were excavated by sinking from the 600 level, a clamshell being used for mucking. The use of 6-ft and 8-ft rock bolts made it unnecessary to timber as the excavation progressed. The cycle followed was drill out, blast, scale down, rock bolt, and muck out. This cycle permitted the use of the muck pile to reach any loose ground opened up by the previous blast. Timbering these pockets would have necessitated special framing and design with resultant higher costs. Also, considerable trouble would have been encountered in placing the forms for the concrete with the timber still in place.

Rock Bolting Development Headings: A large percentage of the development headings in Butte now use rock bolts instead of timber for ground support. Twenty-five hundred feet of the 4400 level at the Mt. Con mine was developed in this manner with an estimated 10,000 tons of waste rock left in place which otherwise would have been broken to make room for regular timber sets.

The wide intersection shown in Fig. 6, part of the vast system of haulageways for Anaconda's new block caving project at the Kelley mine, has been made completely self-supporting by the installation of rock bolts. Timbering an intersection such as this would entail considerably higher costs due to increased material handling and the disposal of extra waste rock broken to make room for this timber. The

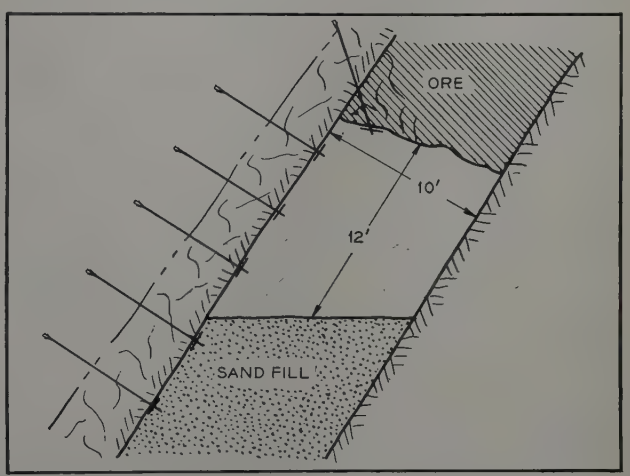


Fig. 4—The rock-bolting procedure used at the Mt. Con mine in stoping by the cut-and-fill method.

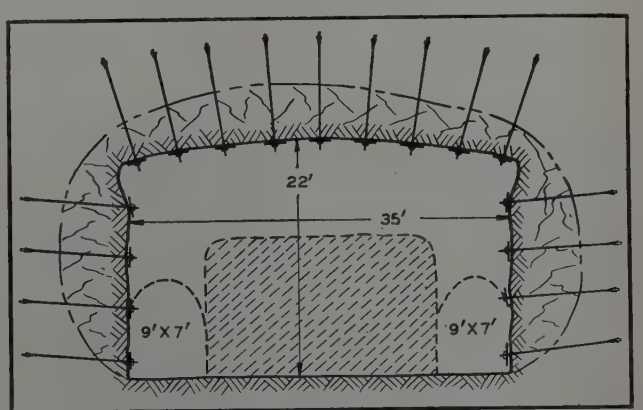


Fig. 5—The rock-bolting method used to excavate an underground hoist room on the 4000 level of the Mt. Con mine.

use of rock bolts makes it possible to drive these headings with the minimum cross-section required for proper clearance of the ore trains. Besides ground support, rock bolts are used to hang trolley lines, pipe, vent tubing, and electrical cable.

Unfortunately, all types of ground in Butte cannot be secured with rock bolts and steel plates alone. Numerous faults, slips, and clay seams at frequent intervals make it necessary to use some means of distributing the support given by the rock bolts over a larger area. War surplus airfield landing mats, shown in Fig. 7, are used for this purpose, as they are easy to install and can be drawn up to conform with the contour of the ground.

Rock bolt sets made of light timber are ideal for repair work in main haulageways not bolted or timbered originally which now require close back and side lagging. They can be installed without interrupting train service, interfering with pipelines, or enlarging to make room for regular timber sets.

These sets, Fig. 8, were installed in 600 ft of haulageway connecting the Badger and Black Rock mines. When the air flow was reversed from fresh to exhaust air, the hitherto firm granite was subjected to rapid alteration at the jointing planes. Overhead safety stringers were advanced as each rock bolt set was installed.

Other Rock Bolt Installations: Rock bolts are ideal for supporting ground in excavations used for housing electrical equipment or storing explosives, since they are fireproof. Timber used in these locations would first have to be covered with wire mesh and then gunited to eliminate the hazard of fire. Fig. 9 illustrates an installation on the 4000 level of the Mt. Con mine housing electrical equipment for a deep level hoist. This room has been open for three years. The ground was badly fractured in places, but there has been no sloughing of the walls or back.

Rock bolts have proved their worth in numerous other underground installations in the Butte mines. These include pumping stations, air conditioning rooms, stations, safety zones, underground repair shops, and locomotive battery change-over rooms.

Besides ground support, rock bolts make convenient hangers for many types of underground equipment. Air and water pipe, electrical cables, see Fig. 10, rigid vent tubing, and ventilating fans are some of the more common uses for this kind of support.

Rock Bolting Practices at Sunshine Mining Co., Kellogg, Idaho

Rocks of the Coeur d'Alene district fall into three groups: 1—Pre-Cambrian rocks of the Belt series; 2—igneous rocks intrusive into the Belt series; 3—unconsolidated sand, gravel, and silt of Tertiary and Quarternary age.

Orebodies of the Sunshine mine occur in shear zones, and along at least one fault, in the north limb of the Big Creek anticline. Upper workings of the mine are in the Wallace formation, and the lowest appear to have entered the Revett. The St. Regis, a thin-bedded argillaceous quartzite lying between the Wallace and Revett, has thus far been the most productive ore-bearing formation of the Silver belt.

Drifts in general strike east and west, the veins and accompanying shearing dipping 70° to the south. The veins occur in shear zones along the steeply dipping north limb of the Big Creek anticline. The bedding in general dips 80° and steeper to the north with occasional slight overturning. The intersecting south-dipping schistosity and north-dipping beds at a maximum angle of 30° produce a weak, slabby, and sometimes blocky hanging wall.

Some four distinct mining methods or modifications of these methods are used at Sunshine in extracting orebodies.

The square-set method is used in orebodies that are 10 ft or greater in width. Stope lengths are generally about 45 ft. The caps are 10x10 in., girts 8x10 in., and posts 9x9 in. All pieces are made from sawed timber. Plan dimensions of each set are 5x5 ft to center line, center line heights being either 6 or 7 ft. Six to seven floors are mined before final cleanout of ore and subsequent filling with waste rock. One floor is left open to begin the next mining cycle. Approximately 46 pct of all ore, except from development headings, is mined by this method.

The method employing caps and stull posts with the caps headed perpendicular to the dip of the vein are used in stopes where the vein is less than 10 ft wide. Cap dimensions may vary from 10x10-in. to 12x12-in. timbers with 6x8-in. post lengths sufficient to give floor heights of 5-ft or 6-ft centers. Caps are headed with 6 to 8 in. of heading. Ore is broken by breasting down. Stope lengths are comparable to the square set. Floor heights and timber dimensions are governed by ground conditions. Here again, six to seven floors are mined before cleaning and filling with waste. The last floor is left open to begin the next cut.

Timbered shrinkage is used where ground conditions are good. This method is comparable to the cap-and-stull system except that 75-ft to 80-ft heights may be mined before cleaning and filling with waste. Back stoping is generally used with this method and stope lengths are also held to 45 ft.

By substitution of rock bolts for timber numerous stopes have been successfully mined on a full shrinkage basis, rock bolts being used to hold the hanging wall and in one instance to bolt the footwall. Stope widths must be no less than 7 ft so a 6-ft bolt, which is standard length for stopes, can be installed.

Early in 1952, through courtesy of the Engineering Research Dept., Anaconda Copper Mining Co., a systematic study was made of methods and techniques used in rock bolting. To date, 12,700 bolts including 11,050 6-ft bolts, 1100 4-ft bolts, and 315 8-ft bolts, together with 235 experimental bolts, have been installed with most gratifying results.

The bolt most commonly used is the 1-in. diam, 6-ft split-wedge type. However, tests have proved that equally good results can be expected from the use of the ¾-in. expansion shield type. These bolts are being used in increasing numbers and no doubt will eventually replace the split-wedge type.

Split-wedge bolts are driven by means of a dolly and driving steel and then tightened to refusal with a 534I-R impact wrench. Fig. 11 shows a member of the rock bolting crew catching up loose ground. The only accessory tool for the expansion shield type is an impact wrench. Most rock bolt holes are drilled with 1⅜-in. insert bits by contractors and regular rock bolt crews, of which there are three currently employed. However, the split-wedge bolt has been used successfully in 1⅝-in. holes.

Now that rock bolts have been used for two years, it is assumed that where a bolt can be firmly anchored it will hold the ground firmly in place. Bolting of unstable rock sections of drifts and crosscuts has been done extensively. Fig. 12 is an illustration.

Numerous stopes have been mined with rock bolts to lend strength to the hanging walls. In driving

Fig. 6—This intersection in a system of haulageways has been made completely self-supporting by the installation of rock bolts.

Fig. 7—War surplus airfield landing mats, shown above, distribute over a larger area the support that is provided by rock bolting.

Fig. 8—Rock bolt sets of light timber are ideal for repairs in main haulageways not bolted or timbered originally.

Fig. 9—Rock bolt installation on 4000 level of Mt. Con mine housing electrical equipment for a deep level hoist.

Fig. 10—As shown here and also in Fig. 12, rock bolts provide convenient hangers for air and water pipes and for electrical cables.

Fig. 11—A member of the rock bolting crew catching up loose ground. Expansion shield bolts are applied with an impact wrench.

Fig. 12—Here, as in Fig. 10, rock bolting serves a dual purpose.

of raises, rock bolts in the hanging wall between the timber pattern have been valuable aids. Shaft stations, grizzly stations, battery stations, and drift and crosscut junctions are bolted as an aid in development and exploration programs. An established practice in crosscut and drift headings is the installation of 4-ft rock bolts from which to suspend rigid ventilating fan lines. Repair work in shafts and winzes is expedited by the use of rock bolts.

There is often a question as to the economic advantages of rock bolting as compared with conventional methods of timbering. With ever increasing labor and material costs, this question is of paramount importance to metal mine operators. The cost, delivered in Kellogg, Idaho, carload lots, of the split wedge and expansion shield bolts is given below, see Table III.

Table III. Comparative Costs of Split-Wedge and Expansion Shield Bolts

Split-Wedge Bolt, 1-In. Diam		Expansion Shield Bolt, ¾-In. Diam	
Length	Cost, $	Length	Cost, $
8-ft bolt with wedge and nut	1.72	6-ft bolt with shield	1.15
6-ft bolt with wedge and nut	1.36	4-ft bolt with shield	0.91
4-ft bolt with wedge and nut	1.04		

Plates 8x8x⅜ in. with 1¼-in. hole are purchased in Spokane at a delivered price of $0.47 each. Angle washers, 15° and 30°, cost $0.22 each.

Recently a crosscut on the 3400 level cut the Yankee Girl vein. After drifting westward for some distance, the back became so weak drift sets had to be installed.

This was done at a total labor and materials cost of $8.51 per linear foot. A comparable section was rock-bolted, using the 6-ft split-wedge type, at a labor and complete bolt cost of $4.82 per ft. This represents a saving of 43.5 pct, not to mention excess rock removed for timbering and the large quantity of heavy timber transported to the working place as compared with the less bulky rock bolts, plates, and wedges, and the uninterrupted advancing of the drift. This drift has since been bolted throughout its entire length. Drifts and cross-cuts that lie in potential rock burst areas have been bolted to impede rock falls. There has been no failure or any observed weakness.

Rock bolting of shrinkage stopes presents an economical approach to ore extraction. As previously stated, 7-ft minimum widths must be maintained. The average width of all the bolted shrinkage stopes has been 11 ft. Stope lengths are held at 45 to 50 ft and stope heights terminate at 90 to 100 ft. Enough ore is drawn after blasting to bolt the hanging wall and back stope or breast down another cut. During blasting the effectiveness of a very small percent of the bolts is lost by breaking of the hard quartzite rock from around the bolt plate. An additional bolt or bolts are installed to replace those damaged. As these stopes are not always bounded on their strike length by raises, a follow-up manway may be timbered as the stope is advanced in height, or the manway may be timbered as the stope is drawn. The adjacent block of ore may then be mined before another raise is driven. Additional bolts, depending on the need, may or may not be installed as the stope is drawn for waste fill. Seven stopes have been mined by this method. Data for five stopes are summarized in Table IV. These five stopes represent a total of 20,608 tons of ore.

Table IV. Summary of Data for Five Stopes

Stope	Tons Per Manshift	Tons Per Rock Bolt	Square Feet of Wall Per Rock Bolt	Cost Per Ton of Installed Rock Bolt, ¢
3700 to 811	12.2	9.7	8.9	36.6
3700 to 910	14.5	12.8	21.1*	27.5
3400 to 14	10.1	18.3	14.0	19.5
3400 to 11	10.8	11.8	12.2	30.4
3400 to 10	7.1	18.4	18.3	19.3
Avg of stopes	10.6	13.8	15.2	25.7

* Footwall and hanging wall bolted.

It may be of interest to compare the above average of the rock bolt shrinkage stopes to a timbered shrinkage stope recently completed. From a timbered shrinkage stope 3991 tons of ore were broken with a productivity of 7.7 tons per manshift. Timber and contract labor cost $0.55 per ton. Slightly more than 20,000 board feet of timber had to be transported to the timbered shrinkage stope. Whereas the average productivity per direct manshift in stoping is 6.8 tons for the mine, the rock bolt shrink stope is nearly 4 tons greater and that of the timbered shrinkage stope approximately 1 ton greater.

It is apparent that use of rock bolts in shrinkage stoping has shown a marked economic advantage of 53 pct over the timbered shrinkage stopes.

Rock bolts have been used extensively in the two cap and three cap development raises. One bolt is driven near the hanging wall heading and one near the center of the set. The pattern appears as the five dots on dice. Occasional bolts in the footwall are

used to stabilize rock that may give trouble. The value of this bolting procedure is readily apparent in repair of raise timbers. One large square-set raise, 20 ft square, was driven from 3700 to 3550. The pattern described was followed on all four walls of the raise. The bolts increased the useful life of this raise severalfold. These raises were driven with stopers using 1⅝-in. insert bits. One-inch diameter slot and wedge type bolts were used. There was no difficulty in obtaining a solid anchor.

Shaft stations, hoist stations, grizzly stations, and battery stations are systematically bolted. Fig. 13 shows a 30x40-ft enlargement for 3700 No. 8 shaft station. These places are bolted to give maximum support rather than to follow fixed patterns. Particular attention is given brows, which develop weakness rapidly. This is especially important in areas where rock bursts may occur.

Since rock bolting was first employed at Sunshine Mining Co. many helpful techniques have been obtained from mining magazines, manufacturers of rock bolts, and Bureau of Mines personnel; however, every mine has unique problems that do not conform with methods employed in other mines. These problems are best worked out by the individual mines to their satisfaction.

Conclusion

In the search for an economic advantage in extraction of orebodies over the increasing costs of labor and supplies, it is of prime importance that established mining methods be supplemented with suitable mining aids to gain this end. Seldom is one unfamiliar with rock bolting impressed by this method, but those using rock bolts give them an important place in their mining operations.

The criterion for judging any new mining technique an improvement over existing methods is: Will it do the same job faster and cheaper? The merits of rock bolting have been proved by this standard.

Fig. 13—Rock bolting in a 30x40-ft enlargement for a shaft station on the 3700 level.

Experience has shown that higher productivity and a more economical unit cost can be realized by the use of rock bolts in shrinkage stopes.

Since rock bolts may be applied immediately to the existing shape of the excavation, rock bolting is faster than timbering, which necessitates framing the timber to conform to local ground conditions. Five rock bolts will normally support as much ground as a set of timber, yet their cost is approximately one half that of the timber. A bundle of 10 rock bolts can be stored or transported underground to the working place with the same effort required for only one piece of timber.

In view of these facts the mining industry, the steel industry, and the U. S. Bureau of Mines have put forth a great deal of effort in promoting rock bolting as a method of ground support. Much progress has already been made and the future will no doubt reveal many new applications which have not yet been considered.

Latest Practice in Burning Cement and Lime In Europe

by O. G. Lellep

Modern shaft kilns in Europe are fully mechanized and burn cement of acceptable quality at 700,000 Btu per bbl and lime at 3.2 million Btu per net ton. Rotary kilns for cement have increased in thermal efficiency by using exit gas heat for preheating incoming raw material and by recovering heat from outgoing clinker in air-quench coolers. The dry process Lepol or ACL kiln has the lowest fuel consumption, 580,000 to 630,000 Btu per bbl, and very low dust loss, about 1 pct weight of clinker. The Holdebank-Gygi system reaches 720,000 Btu per bbl. The calcinator kiln for the wet process consumes 1.1 million Btu per bbl.

IN every country economic circumstances prescribe the method used to produce a commodity at lowest cost. In Pennsylvania a man's wages for working 4 hr buys a ton of coal wholesale; in Germany a laborer must work 40 hr, or ten times as long. In western Europe, therefore, conservation of fuel is of primary importance, and recent practices of burning lime and cement differ from those employed in the United States. This paper presents data collected during a trip through western Europe in 1953.

Considered uneconomical, shaft kilns for burning

O. G. LELLEP Member AIME, is Consultant, Pyro-Processing Section, Allis-Chalmers Manufacturing Co., Milwaukee.

Discussion on this paper, TP 3811H, may be sent (2 copies) to AIME before Sept. 31, 1954. Manuscript, Nov. 11, 1953. New York Meeting, February 1954.

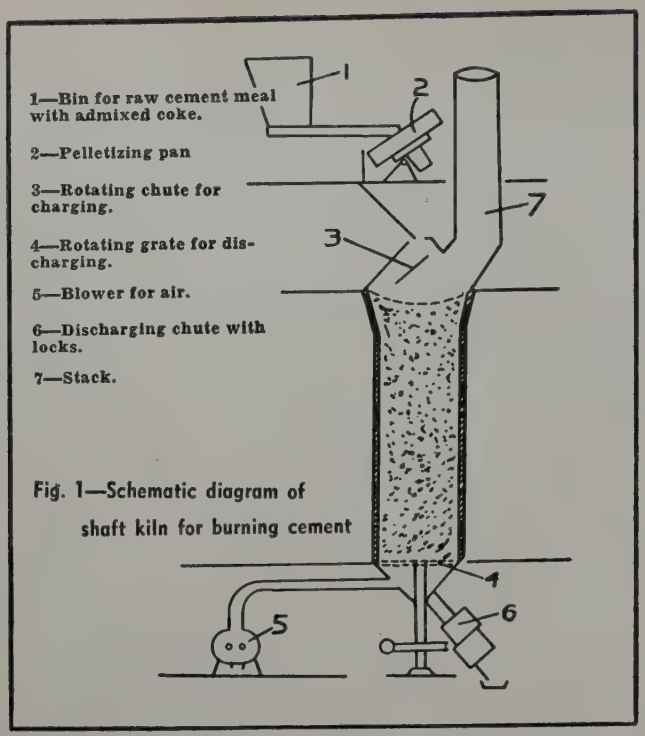


Fig. 1—Schematic diagram of shaft kiln for burning cement

cement disappeared in the United States two generations ago. But because required capital investment is low and operation greatly improved, shaft kilns are still used to some extent in Europe, where fuel costs are high, and are even included in designs for plants under construction. During the last 50 years shaft kilns have been maintained at the same physical size, while their output has been increased at least four times.

The kiln charge, which formerly consisted of large 10-lb briquettes, has been replaced by a relatively small-grained feed of raw meal and crushed coal made in balling drums, or pans, or in pug mills. Positive high-pressure blowers and fully automatic charging and discharging equipment is used. The quality of cement burned in improved shaft kilns meets modern specifications in carefully managed and controlled plants, although a uniformly burned product is harder to obtain in shaft kilns than in rotary kilns, which produce by far the largest part of the general output.

Fig. 1 is a sketch of a modern shaft kiln for cement. The top of a kiln for cement has a conical, widened zone introduced by E. Spohn. Combustion of admixed fuel and a sintering of product is completed mainly in this top section. The charge shrinks considerably during the sintering process and moves downward. The large diameter of unburned material in the zone at the top of the cone diminishes after sintering to the narrower diameter of the shaft and fills the space near the periphery. In older shaft kilns the shaft was cylindrical; consequently after shrinkage due to sintering a free slot was formed near the wall which allowed red hot gases to escape without being utilized. E. Spohn's cone shape has eliminated this defect and the exit gas of a modern shaft kiln in normal operation is below 200°F. Fuel consumption is around 700,000 Btu* per bbl of cement. The usual inside diameter of the cylindrical part is 8 ft and the effective height to which it is filled about 25 ft. Such a kiln can produce 1000 bbl per 24 hr and sometimes more. However, the output of cement shaft kilns remains fairly limited. If the diameter of the shaft is made larger, the uniform distribution of fire and temperature through the wide cross-section becomes difficult and almost impossible to control.

* In Continental Europe heating value of fuels is expressed in lower units, i.e., in net calories. In this report European data are recalculated as gross Btu.

Thermal Efficiency and Rotary Kilns

The rotary kiln is essentially an American development. It is remarkable for its adaptability in burning various mineral, chemical, or ceramic products and today is unsurpassed for high quality, uniformity of burn, and large output. Twenty-five years ago the rotary kiln was extremely wasteful in fuel consumption. Frequently red hot exit gas came out of the stacks and white hot cement clinker was thrown on the clinker pile. Fuel consumption in these older kilns was around 2 million Btu per bbl, or about three times as high as with the best modern practice. During the last generation radical improvements have been made to cut waste of heat at both ends of the rotary kiln. Waste heat at the feed end of the kiln was utilized to produce steam for power generation. Preferably this available heat now is saved to preheat the incoming raw mixture. A rotary drum air-swept clinker cooler has been introduced at the discharge end to cool the white hot product and return the recovered heat as hot air for combustion. Power production from waste heat boilers behind cement kilns is still used in some instances, but the large and more efficient power com-

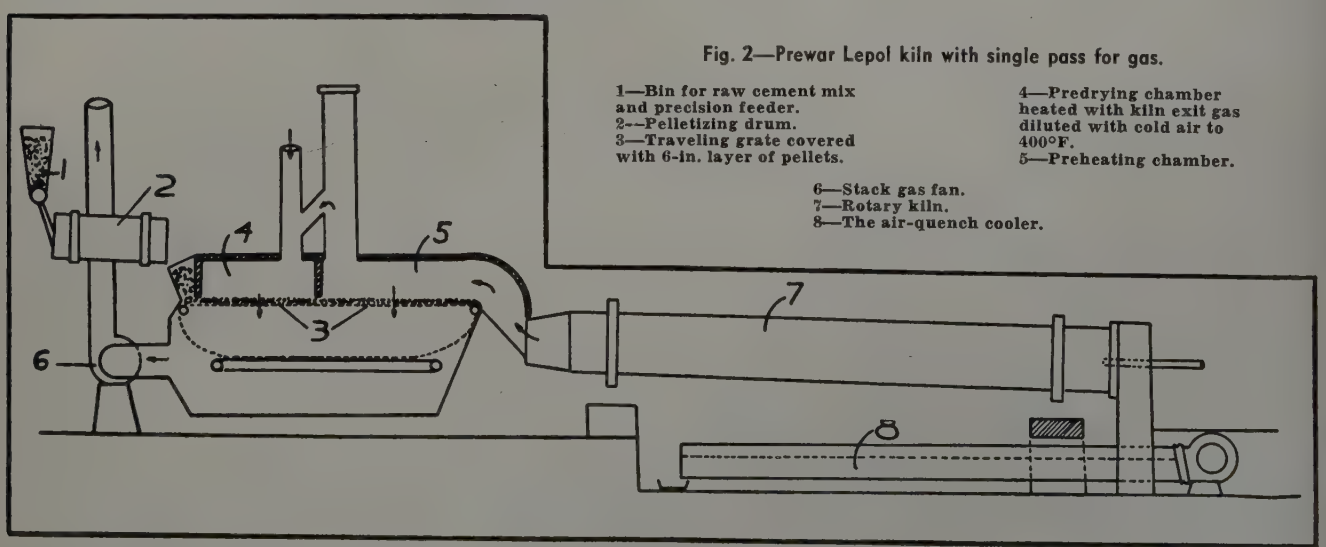

1—Bin for raw cement mix and precision feeder.
2—Pelletizing drum.
3—Traveling grate covered with 6-in. layer of pellets.
4—Predrying chamber heated with kiln exit gas diluted with cold air to 400°F.
5—Preheating chamber.
6—Stack gas fan.
7—Rotary kiln.
8—The air-quench cooler.

Fig. 2—Prewar Lepol kiln with single pass for gas.

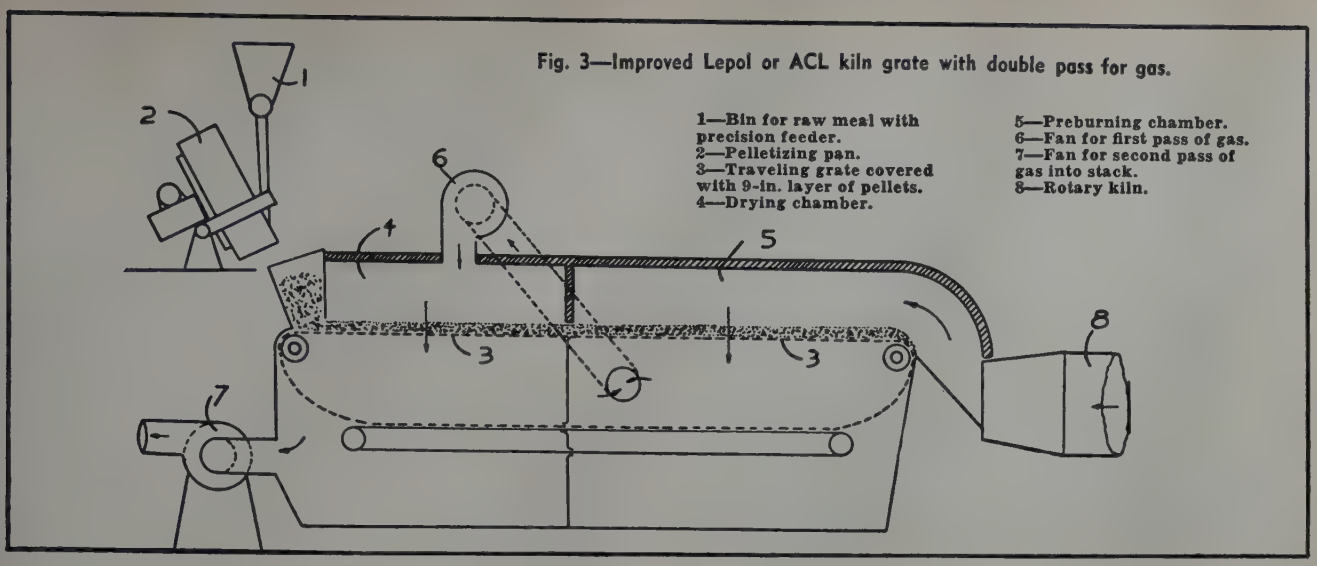


Fig. 3—Improved Lepol or ACL kiln grate with double pass for gas.

1—Bin for raw meal with precision feeder.
2—Pelletizing pan.
3—Traveling grate covered with 9-in. layer of pellets.
4—Drying chamber.
5—Preburning chamber.
6—Fan for first pass of gas.
7—Fan for second pass of gas into stack.
8—Rotary kiln.

panies can usually supply power cheaper than that made from waste heat.

Twenty-three years ago the single-pass prewar Lepol kiln, Fig. 2, was introduced. Instead of generating steam, the exit gas heat is transmitted into a layer of pelletized incoming raw mix and is thus utilized more efficiently. The length of the rotary kiln was cut to from one-half to one-third. Fuel consumption per barrel for dry process cement was decreased to about 700,000 Btu per bbl. One hundred twenty of these kilns were sold in various parts of the world prior to World War II.

This kiln system has been improved in the post-war period, and fuel consumption has been lowered still further. More particulary, passing the kiln gases twice through the layer of pellets has decreased losses radically. Fig. 3 is a schematic diagram of the improved post-war double-pass Lepol or ACL kiln* and Fig. 4 an outside view. The second pass of gas through a layer of moist pellets not only lowers exit gas temperature close to the dew-point of 160°F, but also filters out more dust than a cyclone can filter. During the last 2½ years 23 improved Lepol grate installations have been sold. Table I gives data from four new plants that began operation in Germany and Sweden in 1952 and 1953.

* *Lepol* and *ACL* are trademarks of a kiln of the same system. The Lepol kiln is marketed by Polysius Co., Neubeckum, Germany, and the ACL kiln by Allis-Chalmers Manufacturing Co.

The best consumption in the long modern dry and wet process kilns of conventional design is about 0.8 million Btu per bbl. Output of cement in barrels per cubic foot of internal rotary kiln volume in the Lepol and ACL system is twice or more that in the kilns of conventional design.

The dust from dry process cement kilns without expensive Cottrell dust precipitators usually varies between 3 to 10 pct of the weight of clinker. The improved Lepol kilns employ a dust cyclone after the first pass of gas. No cyclone is normally used after the second pass or before the stack. After the second pass in the ACL kiln system where the exit gas is filtered through a layer of moist cement pellets the dust averages 1 pct of the weight of clinker. Thus a double-pass ACL grate not only saves heat, but also collects dust more efficiently than a cyclone.

Normally the wet process cement slurry of non-colloidal materials can be filtered rapidly to about 17 pct moisture on a drum-type filter. In exceptional cases it is possible to lower the moisture content to 14.5 or 15 pct moisture by beating the filter cake on the drum with so-called flappers. Such a filter cake can be pelletized and burned in the Lepol (ACL) kiln system.[1]

One double-pass Lepol plant reports a power consumption of 2.66 kw-hr per bbl of cement produced, including coal grinding. Operating time in another plant has been 90 to 95 pct of ideal plant operating time. Fig. 4 is a view of the Lepol installation, which went in operation in 1953.

The calcinator, built by the Miag Co. in Germany, is used in large numbers on wet process kilns and also to some extent on dry process kilns to reduce heat consumption. It is a heat-exchanger consisting of a slowly rotating drum, 13 ft in diam by 10 ft long, with a perforated cylindrical shell, and is filled with large pieces of rolling heat-exchanger members, such as cutoff pieces of 4-in. pipe, or I-beams. The hot exit gas is passed through the rolling layer of heat-exchange bodies while the cement slurry is sprayed on the top surface of the rolling heat-exchanger.

E. Schott,[2] Table II, reports the following data about a wet process calcinator, 13 ft in diam by 10 ft long. The kiln is 10.5 x 175 ft long.

Table I. Operation Data for Four Lepol Kiln Plants in Germany and Sweden

Designation of Installation	Rated Output, Bbl Per 24 Hr	Average Output Over 5-Month Period, Bbls Per 24 Hr	Heat Consumption Measured During a 24-Hr Test Period, Btu Per Bbl	Average Heat Consumption Over a 5-Month Period, Btu Per Bbl	Reported Dust Loss into Stack, Pct from Clinker
1	3000	3480	595,000	630,000	1.5
2	3000	3400	570,000	600,000	Very little
3	1800	2100	580,000	Below 630,000	0.81
4	1800	No data yet	595,000	No data yet	0.7

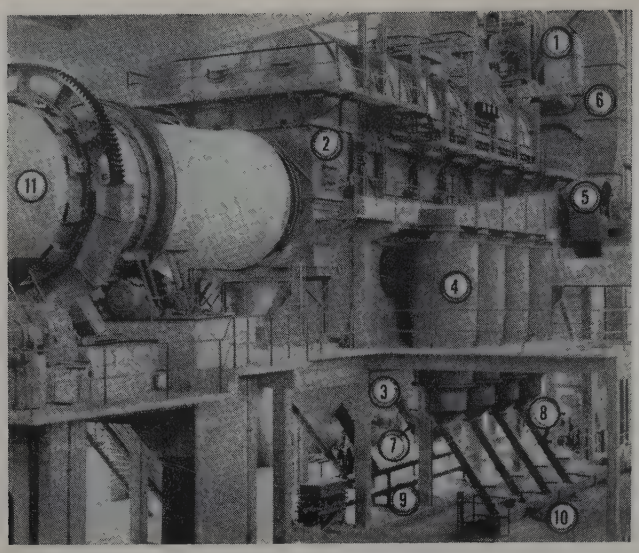


Fig. 4—View of the two-pass Lepol grate built by Polysius Co.

1—Balling or pelletizing pan.
2—Upper part of furnace.
3—Lower part of gas chambers built of concrete.
4—Cyclone for dust after first pass of gas through the pellets.
5—Fan for first pass of gas.
6—Gas duct leading into the predrying chamber.
7,8,9, and 10—Enclosed chutes and transporting devices taking dust into rotary kiln.
11—Rotary kiln.

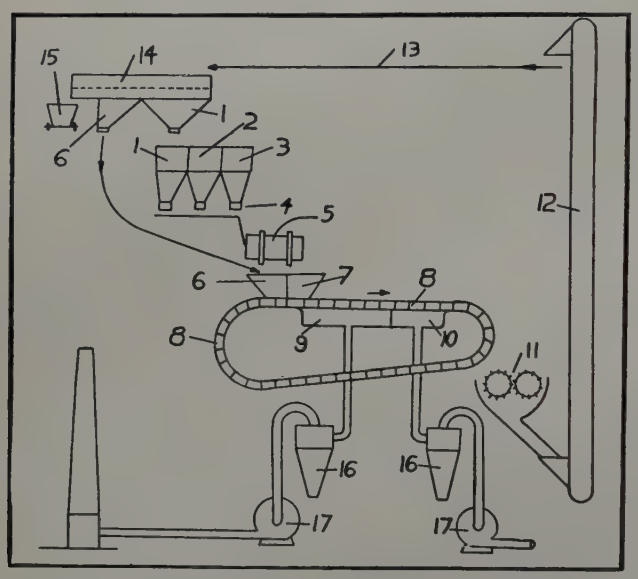


Fig. 5—The Lurgi-Wendeborn sintering process for cement.

1—Bin for recirculated burned fines.
2—Bin for raw meal.
3—Bin for coke breeze.
4—Automatic proportioning scales.
5—Pelletizing drum.
6—Screened clinker to protect grate.
7—Bin for pelletized feed.
8—Sintering machine.
9—Suction box for stack gas.
10—Suction box for hot gas used for predrying raw material.
11—Crusher.
12—Elevator.
13—Conveyor.
14—Screen.
15—Good, coarse clinker.
16—Cyclones.
17—Fans.

Mention should be made of the Kloeckner-Humboldt cyclone heat-exchanger for dry process kilns; four kilns of this type were operating in Germany in the summer of 1953.

Table II. Data for Wet Process Calculator

Size of Calcinator: 13-Ft Diam x 10 Ft Long	Kiln Output, Bbls Per 24 Hr	Exit Gas Temperature, °F	Heat Consumption, Btu Per Bbl
Before installation of calcinator	1920	1400	1,510,000
After installation of the calcinator	2000	270	1,116,000

The Holderbank-Gygi heat-exchanger was developed in Switzerland over 10 years ago. A few are running in that country and two in Sweden, but none in Germany. Tham and Sylvan[3] of Skanska Cement Co. have reported on this system. To improve heat transmission by conduction from the hot gas to the raw mix inside the feed end of the kiln, the gas is caused to rotate at a higher velocity in a spiral movement. Four turbine-type windmill wheels of heat-resisting metal are fixed to the inside wall of the kiln. Lifters are bolted to the inside kiln wall between the turbine wheels. They lift the nodulized raw mixture and drop it through the hot gases, thus increasing the heat-receiving surface of the material to be heated. Two older wet process kilns have been converted to this system in Sweden; Table III shows the dimensions, outputs, and fuel consumption.

The lifting and dropping of pellets in this heat-exchanger causes considerable dust, which is precipitated and re-utilized in the Lepol system. Besides the improved rotary clinker cooler used on most prewar Lepol kilns, modern kilns preferably use one of three well-known air-quenching coolers.

The process of burning cement on sintering machines, invented by an American, R. W. Hyde[4], was developed through long years of work in Germany by H. Wendeborn[5] of the Lurgi Co., successfully operated by K. Boerner, and further improved by R. Rohrbach.[6] Fig. 5 is a diagram of a Lurgi cement-burning plant with a sintering machine. Numerous tests have shown that a more acceptable product results if in addition to raw cement mixture and crushed coke 30 to 60 pct of feed to the machine is already burned clinker. An accurate proportioning between the raw mixture and the fuel is important, requiring reliable automatic scales. It appears difficult always to burn the layer on the grate to a perfectly and uniformly well-burned clinker, an objective relatively easy to obtain in a rotary kiln. Therefore it is necessary to crush the burned layer and screen out the underburned part, which is returned into the circuit and mixed with the raw feed. To prevent overheating, the grate bars have to be protected with a layer of already burned clinker.

According to Rohrbach[6] a plant with an active grate area, 6.5 ft wide by 43 ft long, produces 2000 bbl per 24 hr. Toal heat consumption in the ignition

Table III. Data on Conversion of Wet Process Kilns to Dry Process Holderbank System

No. of Kiln	Length of Kiln Including Cooler, Ft	Diameter of Kiln: Feed End	Diameter of Kiln: Burning Zone	Output When Run on Wet Process, Bbl Per 24 Hr	Output After Conversion to Dry Process Holderbank System 24-Hr Test, Bbl Per 24 Hr	Heat Consumption
1	290	9 ft 4 in.	8 ft 5 in. x 10 in.	1600	1950 est.	720,000
2	290	10 ft 5 in.	9 ft x 10 in.	2200	2450	actual input

flame and admixed carbon amounts to 810,000 Btu per bbl over long periods of operation. Good cement quality has been produced by this burning equipment, provided the equipment is well maintained, supervised, and controlled.

Lime and Dolomite Burning

Because of high fuel cost, lime is burned in shaft kilns only in Germany and France. Rheinische Kalkwerke at Wuelfrath has a set of six shaft kilns of the Seeger design, apparently operated at an excellent fuel efficiency, see Fig. 6. Inside diameter is 12.8 ft and wall thickness 20 in. Wall thickness of six new shaft kilns of improved design has been increased to 30 in. by means of insulation. The cylinder is filled with stone and lime to a height of 57 ft. Two sizes of stone are burned: 2⅜ to 4¾ in. and 4¾ to 7 in. Output for the smaller size of stone is 135 tons per 24 hr per kiln and for the larger size 120 tons. Charging and discharging is fully mechanized. Three men can operate six kilns if they need not be concerned with stone supply.

To avoid segregation, particular attention is paid to the uniform distribution of stone and coke in these mixed feed kilns. Water-gage pressure required to move the gases through the kiln varies from 2.35 to 4 in. The results obtained are remarkable. The lime is discharged cold and the top gas temperature is only 212°F. The gas analyzer registers a steady 41 pct carbon dioxide and a scarcely noticeable carbon monoxide content. Heat consumption is reported as only 3.2 million Btu per net ton of lime burned.

In the older system of charging these kilns the skips of stone were charged on top of the kiln, and thereafter a separate skipload of coke was distributed on top of stone inside the kiln. It was difficult to distribute coke uniformly on the layer of stone because of poor visibility inside the kiln. In the new system of charging the round, the rotatable skip for charging is first filled with stone while on the ground floor, and thereafter a uniform layer of coke is very carefully and evenly distributed on top of the stone in the skip on the ground floor. There the operator can see and control the fuel distribution.

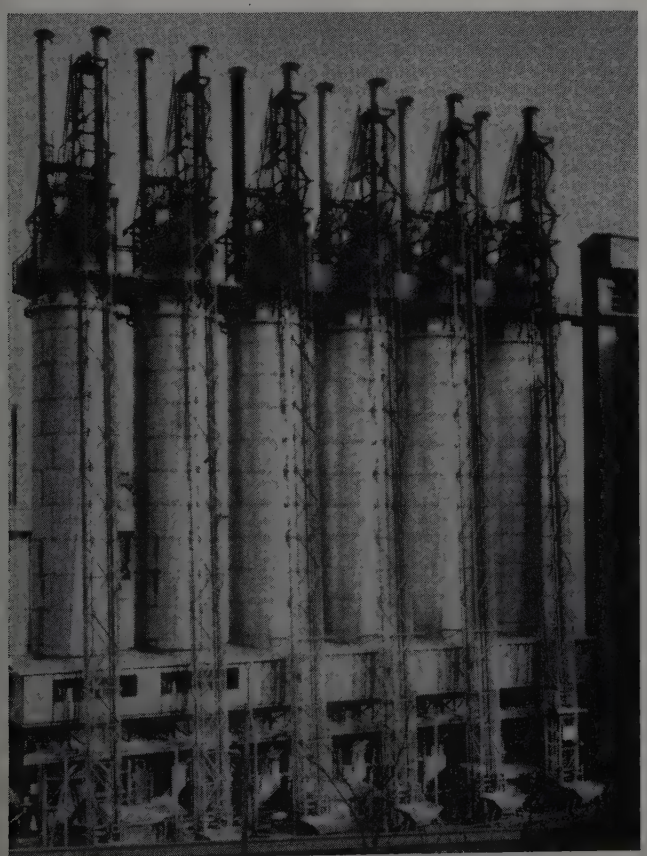


Fig. 6—A group of highly efficient fully mechanized lime kilns consuming only 3.25 million Btu per ton of lime. Rheinische Kalkwerke at Wuelfrat, Germany.

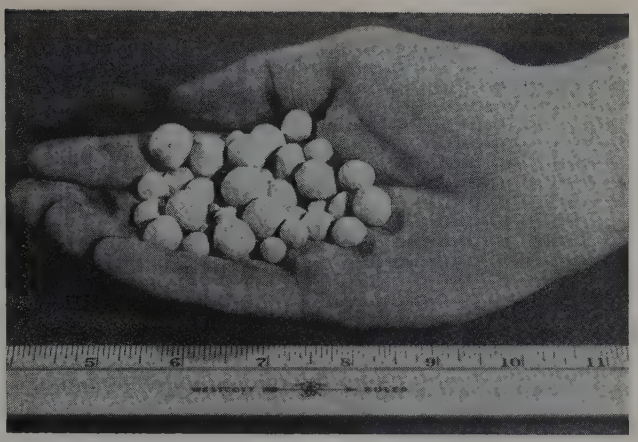


Fig. 7—Shape and relative size of pellets formed in pelletizing drum or pan of cement raw mixture.

The largest part of the dead burned dolomite required as refractory material in the steel mills is still burned in mixed fuel-fired shaft kilns. Limestone spalls, or stone from 2 to ¼ in., cannot be burned efficiently and with sufficient output in a normal shaft kiln. The known authority on industrial furnaces, W. Heiligenstaedt, is building a patented gas-fired shaft kiln for this purpose. It has two narrow rectangular stone and lime-filled shafts, each 26 ft high by 17 ft long and only 25 in. wide. The gases are recirculated crosscurrent instead of countercurrent, in three passes through the thin layer of stone. Estimated output is 100 metric tons per 24 hr.

Conclusions

Owing to war damage and lack of capital most cement and lime plants in western continental Europe need modernization, but a number of plants mentioned in this report remain the leaders in efficient fuel utilization and competent management.

The above data were obtained from sources considered reliable; however, the writer cannot be responsible for their accuracy.

References

[1] Both the Lepol and ACL systems are based on O. G. Lellep patents.

[2] E. Schott: *Test of Cement Burning Equipment Used in Germany*, pp. 117-175. Report presented at Meeting of German Portland Cement Association, May 5, 1953.

[3] L. H. Tham and P. Sylvan: Producing Cement Clinker at Less than 700,000 Btu per Bbl. *Rock Products* (September 1953) pp. 78-87, 124-126.

[4] R. W. Hyde: U. S. Patent 2,090,868.

[5] H. Wendeborn: Theory and Practice of Burning Cement Clinker on the Sintering Machine. *Zement-Kalk-Gips* (January 1949).

[6] R. Rohrbach: Burning Cement on Sintering Machine. *Toindustrie Zeitung* (1953) Heft 9/10, pp. 157-160.

[7] W. Eitel et al: New Cement Burning Process. *Rock Products* (March 1932), pp. 42-47.

[8] A. Bellwinkel: Rotary Kiln Tests. *Zement-Kalk-Gips* (September 1953) p. 331.

[9] B. Helming: Development of the Lepol Kiln. *Zement-Kalk-Gips* (June 1953) pp. 210-218.

Application of Closed-Circuit TV to Conveyor And Mining Operations

by G. H. Wilson

INTRODUCED in 1946 to serve a need in power-plant operation, closed-circuit TV has been used by well over 200 organizations in approximately 25 different industries. Known as industrial television, or simply ITV, it can be described as a private system wherein the television signal is restricted in distribution, usually by confinement within co-axial cable that directly connects the TV camera to one or several monitors, Figs. 1, 2. The picture is continuous and transmission is instantaneous, permitting an observer to see an operation that may be too distant, too inaccessible, or too dangerous to be viewed directly. Destructive testing or the machining of high explosives can now be conducted hundreds of feet away by personnel who still have close control through the eyes of the TV camera. It is also possible for one man to control operations formerly requiring the co-ordinated efforts of several workers. For example, at a large midwestern cement plant conveyance of limestone from primary crusher to raw mill and loading into five storage bins once necessitated the work of two men, one having little to do but prevent spilling of material by manually moving the tripper on the belt conveyor as occasion required. TV cameras mounted on the tripper now provide bin level indication to the conveyor operator at the crusher position so he is able to control the entire loading operation remotely, Fig. 3. By means of a switch, the picture from either camera is alternately available on a single viewer, or monitor, Fig. 4. Each camera is mounted on the tripper by means of a simple adjustable support and looks down into the bin, which is identified by the number of cross members on the vertical rod. Each associated power unit is located on a platform above the camera, Fig. 5. This centralized control by means of TV often has produced superior results, and in many instances saving in operating costs has been sufficient to write off equipment costs within six months to a year.

Where a key portion of a process may be enclosed or otherwise inaccessible, TV again reduces the likelihood of mistakes and permits closer control by making available to the operator valuable information he might otherwise never possess. An example of this can be found at a strip mine where the coal seam lies 50 ft or more below the overburden, which is removed by a large wheel shovel. From his centrally located position the shovel operator was unable to judge accurately to what extent the wheel buckets engaged the earth. His chief indication of efficiency was the amount of overburden on the belt conveyor as it passed his control point 75 ft from the wheel. Now, two television cameras mounted on the tip of the boom permit the operator to view the wheel from each side and provide him with a close-up view of the buckets so that he can take immediate and continuous advantage of their capacity, quickly compensating for ground irregularities and avoiding obstructions, Fig. 6.

G. H. WILSON is Supervisor of Electronic Sales, Diamond Power Specialty Corp., Lancaster, Pa.

Discussion on this paper, TP 3812B, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Jan. 4, 1954. New York Meeting, February 1954.

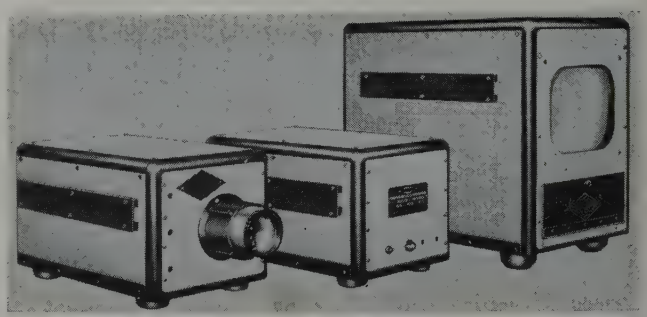


Fig. 1—Basic industrial television equipment: left to right, TV camera, associated power unit, and viewer or monitor. Equipment is designed for continuous operation.

While the word *television* conjures up visions of highly complex and intricate apparatus such as that employed in modern TV studios and transmitting stations, the term *industrial television* should indicate compact, straightforward equipment. Most present-day ITV systems contain fewer than 25 tubes including camera and picture tubes. The average home television receiver alone requires at least that many tubes.

Equipment like that illustrated in Fig. 1 contains only 17 tubes, of which 3 are in the camera. It can operate continuously and dependably, without protection, in any temperature from 0° to 150°F. It consumes less current than a toaster and weighs under 140 lb. Camera and monitor may be separated by 1500 to 2000 ft and by greater distance with additional amplification. This equipment is designed to withstand vibrations up to ±1/16 in. and will operate successfully under more severe conditions of vibration and heat when suitable enclosures are provided. Any number of cameras may be switched to a single monitor, and any number of monitors, within reason, used simultaneously.

Two types of applications in the mining industry have already been described. A third under serious consideration by several organizations will make use of ITV for remote observation of conveyor transfer points at copper concentrating plants so that evidence of belt breakdown and plugging of transfer chutes can be spotted immediately and costly overflow of material avoided.

A television camera will soon be installed to view a trough conveyor near the exit of an iron-ore crusher to indicate clogging of the crusher as evidenced by reduction or absence of material on the

belt. Other means, both electrical and mechanical, have been tried without success.

Another mining organization is concerned with the potential danger to personnel which exists in connection with remotely operated manskips. Engineers have suggested use of a television camera in the cage to let the operator know unmistakably when it is safe to put the hoist in motion without danger to personnel.

In a mine shaft a TV camera has been installed viewing two sheave wheels. This provides the control operator 1500 ft away with a positive and safe means of knowing when cables disengage from the wheels, enabling him to stop operations in time to avoid an accident and prevent damage to equipment. The need for observation is sufficiently critical to have required direct observation by an individual whom the camera has relieved for productive work.

Demonstration has proved that closed-circuit TV can be used successfully to view the interiors of cement kilns. Its use will permit remote control of 6 to 10 kilns by one highly trained operator, greatly facilitating product uniformity, improving working conditions, and at the same time reducing operational costs.

It is reasonable to conclude that the current trend toward use of remotely controlled drilling equipment will eventually give rise to the need for ITV so that proper visual control can be afforded. Although engineering difficulties are numerous, several organizations have become interested in the possibilities.

To evaluate more fully the feasibility of television for a given application it is essential to know the capabilities and limitations of the several types of camera tubes currently available for ITV equipment. As the camera tube is the heart of any TV system, its capabilities and limitations become, in a general sense, those of the system itself.

The image dissector camera tube was the first tube used for this purpose. Because it contains no filament or electron gun, or in other words because it works on the *cold-cathode* principle, it has an extremely long life and is guaranteed for 8750 hr of operation. Image dissectors installed as far back as 1947 are in continuous operation today even after 54,000 hr of service. The dissector, illustrated in Fig. 7, has limited sensitivity, however, and at least

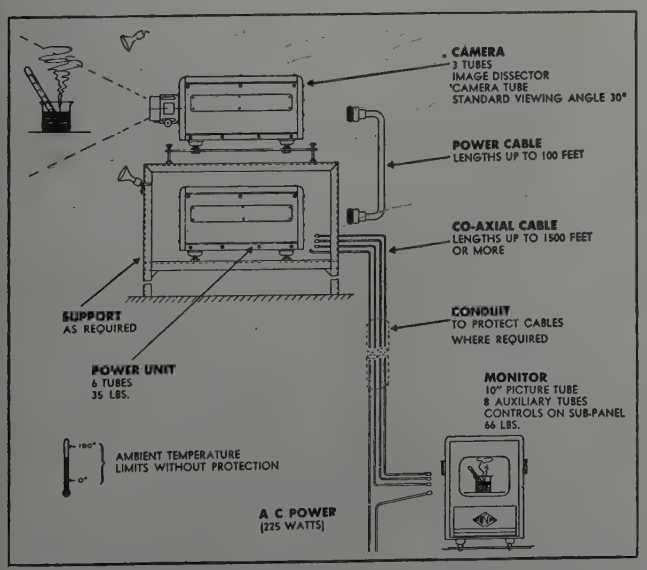


Fig. 2—Typical industrial television installation showing interconnection of components.

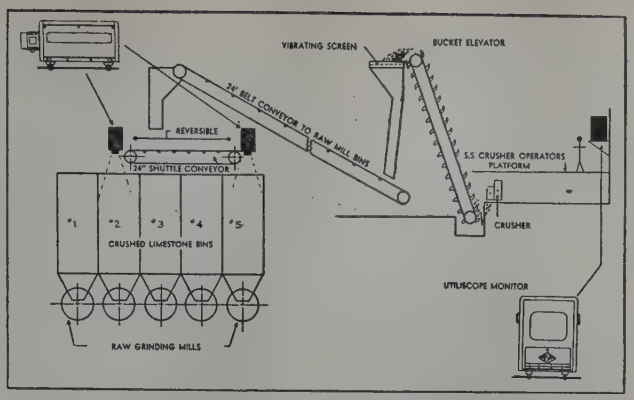


Fig. 3—Diagram above shows how it is possible for one operator to load limestone in storage bins by remote control.

150 foot-candles of illumination are required for a satisfactory picture. While it is not currently practical to design this tube to produce a high-definition picture, it is capable of producing sufficient detail for most industrial applications encountered thus far. Further, since it is a cold-cathode tube it is highly stable, requiring only slight adjustments, and is especially suitable for applications involving continuous operation. Because it is free from the *memory effect* encountered in other types of camera tubes, it is well suited to applications where rapid motion is encountered. Its freedom from *blooming* becomes an important factor where high light levels or sudden surges of light are encountered, as in various types of industrial furnaces.

A more recent introduction is the Vidicon camera tube, diam 1 in. and length 6¼ in. The Vidicon is capable of producing a television picture having greater detail than that produced by the dissector. Although it is subject to memory effect when light levels are low, its sensitivity is greater than that of the dissector to the extent that it can produce acceptable pictures with illumination as low as 25 ft-c, even as low as 10 ft-c in some instances. Life expectancy of the Vidicon is tentatively considered to be from 2000 to 4000 hr. It is currently guaranteed for only 250 hr, however, and for all but the first 15 hr of that total on a pro-rate basis.

Recently several other types of camera tubes have been announced, all operating on the same principles as the Vidicon, with similar capacities.

The image Orthicon, long the only tube used in TV studio work, is too critical and its associated equipment too complex to be considered for closed-circuit industrial work. Although it is eye-sensitive and can produce acceptable pictures at light levels as low as 1 ft-c, its life expectancy of approximately 1200 hr would make operating costs prohibitive for general industrial application.

From this it becomes apparent that type of service, severity of operating conditions, available illumination, and definition requirements are the main factors leading to the choice of a system.

Since the TV camera must often be located at a point where dust, dirt, heat, and vibration, either separately or in combination, are abnormally severe, protective devices usually become an important part of the installation. Many types have been designed to provide adequate protection under the wide range of conditions encountered thus far.

Considerable work has been required to develop special windows which shield furnace-viewing cameras against temperatures approaching 2500°F. Dustproof and explosion-proof housings have been

Fig. 4—Bin level information is received on television screen by crusher operator who controls loading mechanism remotely.

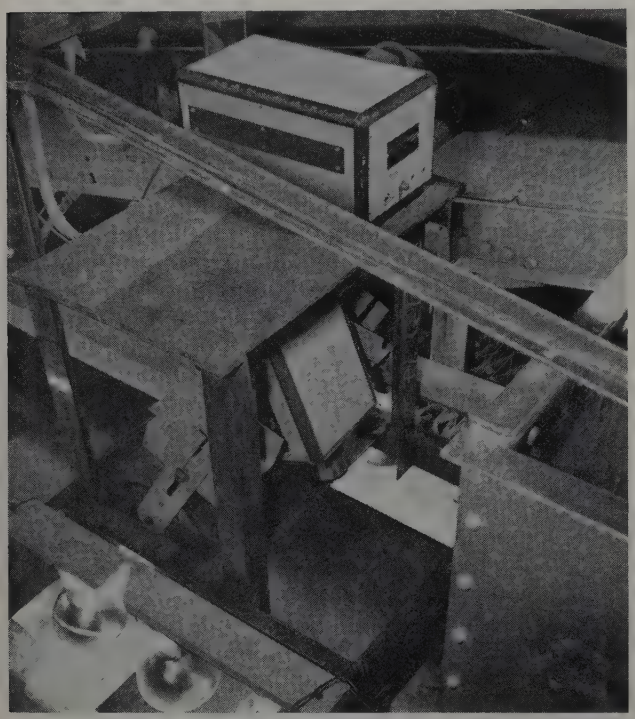


Fig. 5—Camera views area underneath tripper, providing picture of limestone level in bin to remote control operator.

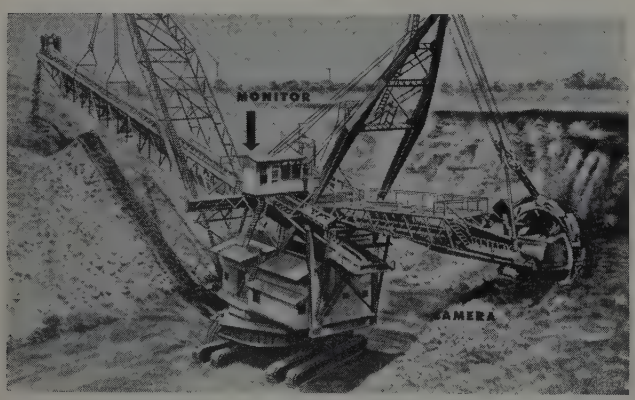


Fig. 6—Television camera mounted near scoop of earth remover provides close-up view of digging operation to operator in cab. Armored housing is removed to show camera.

designed to meet rigid requirements. Weatherproof housings protect cameras in outdoor locations and include several interesting design features to meet unique requirements.

Protection against wide variations in temperature is provided automatically by thermostatic actuation of a blower or heating element whenever pre-set ambient temperature limits are exceeded. Providing optimum visibility under adverse conditions requires inclusion of additional accessories originally designed for the automotive industry. A defroster for the observation window, remotely controlled, works in conjunction with a truck-type electric windshield wiper, also controlled from the viewing position, to permit observation under conditions of rain, snow, or sleet.

In some instances one camera can do the work of two by utilizing a turret containing several types of lenses that can be switched and focused from the monitor position. Camera accessibility and the relative need for observing two or more points simultaneously will determine the justification for such arrangements. Remotely controlled camera tilting and traversing supports increase its utility.

When use of ITV is under consideration, it is particularly important to determine exactly what must be seen by the camera. Simply to decide that the camera should view a belt conveyor, for instance, is insufficient. The key to obtaining desired results might depend on determining the presence or absence of material on the conveyor, amount or rate of spillage at a transfer point, size of material particles, or changes in the appearance of the material, to cite a few possibilities. Once the viewing requirement has been determined, camera location should be considered. In some instances the most obvious locations cannot be used because of interfering structures, or conversely because no structure is available on which the camera can be supported conveniently. Final choice of camera location dictates the type of lens required and often the need for accessory equipment. As the position of the camera varies so does its accessibility, ease of installation and, to some degree, the conditions under which it operates, all important considerations for the supplier who is suggesting the type of equipment and accessories for optimum performance.

Some viewing requirements which seem easy to the human eye may be difficult for the TV camera and vice versa. While the eye is more sensitive than the camera tubes used in ITV equipment and can see much greater detail normally, the camera lens may take in wide areas at a single glance, whereas the eye must scan them. One type of tube is sensitive in the infra-red and therefore sometimes obtains a more informative picture of incandescent bodies than is possible for the human eye. Fig. 8 shows the spectral response curves for Vidicon and image dissector as compared to the human eye.

Maintenance of closed-circuit TV equipment of the kind illustrated consists mainly of cleaning the lens and camera tube surfaces. Frequency of cleaning will be governed by conditions under which the camera must operate, but a definite schedule should be maintained, probably on a weekly basis under average conditions. Dust accumulation in other parts should be removed once a month, although this equipment has withstood prolonged neglect.

The 15 auxiliary tubes should be replaced at least every six months, not only when tubes fail, but on a definite schedule if application is critical. Since the

Fig. 7—The image dissector camera tube, heart of the Utiliscope ITV system. Internal parts of the tube are shown to left and right.

combined worth of auxiliary tubes, at list prices, is less than $45.00, their replacement after 4500 hr of service represents an insignificant hourly operational cost. All are commercially obtainable, as is the picture tube in the monitor which has a life expectancy of about one year and replacement cost of less than $35.00.

Camera tube replacement cost is a major consideration. Based on reasonable life expectancy (6 years) of the image dissector, priced at $1210.00,

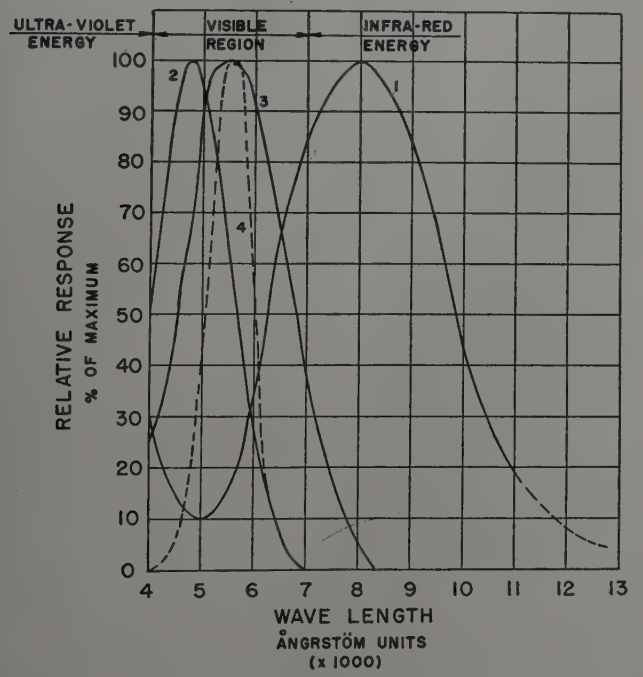


Fig. 8—Spectral response curves for image dissector and Vidicon camera tubes. Curve 1—image dissector, red sensitive; Curve 2—image dissector, blue sensitive; Curve 3—Vidicon, 6198; Curve 4—human eye.

this cost would be $200.00 yearly. Normally the dissector is guaranteed for one year, but it can be covered alternately by a two-year pro-rated use arrangement made possible by the record of service to date. Use of the Vidicon tube, also available with the type of equipment illustrated, will involve higher replacement cost, despite lower initial cost, because of much shorter life expectancy. It will be apparent that operational cost with either tube is nominal.

Because ITV equipment such as that illustrated is of straightforward design and requires no critical

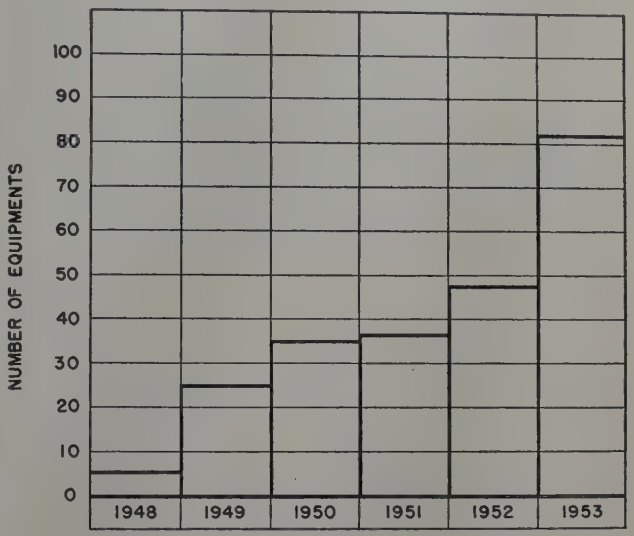


Fig. 9—Installations of ITV equipment. Yearly totals from 1948 to 1954.

alignment procedures for proper performance, service can be successfully accomplished by electrical maintenance personnel within an organization. It is essential, however, that such personnel understand the operation of the equipment. Sufficient time is allotted for this by the manufacturer's service representative when the equipment is adjusted initially. It is seldom that an organization having use for ITV equipment does not utilize sufficient instrumentation of other types to warrant the employment of electrical technicians.

The gradual growth of ITV during the past three years is indicated by Fig. 9, which shows the number of installations yearly from 1948 to 1954. In the average case at least six months elapsed between sale and installation, chiefly because many equipments were purchased for plants or facilities then under construction. The average elapsed operating time of all equipments, 95 pct of which were serving applications requiring continuous operation, was 2.2 years or 19,250 hr. Thus by 1954 ITV had provided more than 500 equipment-years of service to industry, a total indicating that it had progressed well past the point of experimental operation and become established as a means of instrumentation. Data is provided for only one manufacturer, as no other was active in this field throughout the period represented. Additional installations by other suppliers during part of this period were not sufficient to alter the trend significantly.

In view of its extraordinary commercial growth, which will continue unabated for at least a decade, it may be difficult at first to conceive that employment of TV for entertainment purposes could be outstripped by its expansion in closed-circuit usage, as has been predicted. However, it must be realized that its use as an entertainment medium has obscured appreciation of its fundamental purpose. Basically television is remote vision, as its definition implies, and while ITV is far from maturity, it has progressed sufficiently to warrant serious consideration as a medium of great industrial accomplishment.

The Georgetown preparation plant of Hanna Coal Co.

Oil Spraying at the Georgetown Preparation Plant

by A. F. Meger

Coal treatment by oil spraying receives special attention at the Georgetown plant for the dividends it pays in satisfying present customers and attracting new markets. Customer satisfaction requires care, quality control, and use of suitable oils.

REPRESENTED here are the ideas and varied experiences of many people in the Hanna Coal Co. who have helped develop, over a number of years, an efficient and flexible method for spraying controlled amounts of oil on coal. Since the Georgetown preparation plant began operations early in 1951, oil treatment of coal has received special attention. It is believed that oil treating will satisfy present customers and also attract new markets.

The first step in developing the new method of oil spraying was the construction of a dust cabinet for testing 50-lb samples of treated coal with the available oil sprays on the market. Tests results were tabulated for both indoor and outdoor storage.

The graph, Fig. 1, shows results of the original tests made with a 700-sec residual blend and a 500-sec lubricating oil on Georgetown stoker coal of 1¼x⅜-in. size.* The advantages of better price, smaller required amounts, and better storage characteristics originally credited to residuals have been refuted by results of later tests made with accurate metering equipment. Residuals still give the price advantage, but it has not been possible to arrive consistently at the other two benefits, so for all practical purposes residuals and pale oils are now classed together for required amounts and for effective storage time, see Fig. 2 and Table I.

The one major disadvantage of using residuals is the odor. Masking agents may help, but one load improperly blended can raise havoc. Some pale oils also, although odorless when delivered, give off a sweet sickening odor when atomized under pressure. It is recommended that all products be tested before they are used.

There is a specific place for all spraying oils. For example, at a power plant a severe dust condition exists around the stockpile area and in parts of the plant. The cheapest spraying oil made, regardless of odor, will do an excellent job in this case and will save the company 3 to 5¢ per gal or more. Domestic trade, on the other hand, must be carefully handled to prevent complaints about odor, and the only way to do this is to stay with the proved products after the odor tests have been made.

The preparation plant at Georgetown has facilities to handle either tank car or tank truck deliveries. Steam is available at the unloading point if the weather is severe, and there is an indicator at the unloading point to prevent overflowing the 20,000-gal underground storage tank. Systematic test samples are removed from each delivery and sent to the lubrication engineer to insure quality control.

Fig. 3 illustrates the chutes in which smaller sizes of coal are treated. Fig. 4 shows the entire spraying system. Buried in the ground is a 20,000-gal tank with immersion heaters grouped around the feeder line to insure adequate gravity flow to the pump under all conditions of weather.

Oil flows to a 500-gal preheating tank where the temperature is kept about 150°F by six 1½-kw immersion heaters thermostatically controlled. Hot oil is fed through a self-cleaning disc-type filter to the hydraulic pump. Valves and piping make it possible to transfer oil from one tank to another or to drain the lines for maintenance purposes. All this equipment is below ground level in a covered pump room,

* All information contained in this report deals only with the Pittsburgh No. 8 seam of bituminous coal.

A. F. MEGER is Maintenance Engineer, Hanna Coal Co., Division of Pittsburgh Consolidation Coal Co., Adena, Ohio.

Discussion on this paper, TP 3558F, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Oct. 22, 1952. AIME-ASME Fuels Conference, Philadelphia, 1952.

protected by CO_2 extinguishers and kept dry by an oversize sump pump.

The operating pressure, 750 to 1000 psi, is maintained at all times by a system of relief valves, solenoid valves, and time delay and pressure switches. As this entire plant is operated by only five control men, it was necessary to have a system as completely automatic as possible. A similar setup could be installed without the automatic devices, on as large or small a scale as desired.

Table I. Typical Screen Analysis of —⅜ Coal

Screen	Pct
⅜x¼-in.	24.3
¼x4-in.	17.2
+6	15.1
+8	10.2
+14	13.1
+24	7.5
+48	4.3
+100	2.9
+200	1.8
+325	1.1
—325	2.5

The operator controlling the five loading booms receives instructions to treat a specified number of cars. All he must do is push the start button and watch a gage until operating pressure is reached. The automatic circuit now takes over the controls. Oil starts through the lines, preheated when necessary by resistance heating, the 1-in. pipe being used as the heating element. All pipe is insulated both for heat and electrical conductivity. If the weather is extremely cold and oil is sluggish, the pressure will immediately jump to 1100 lb, the pressure setting of the safety relief valve. A pressure switch will now activate a solenoid valve and timer, opening a through line for cold oil to return to the preheat tank and for 150° oil to fill the circuit for 20 sec. The timer then closes the solenoid valve and routes the oil through the proper relief valve directly to the pump. This relief valve is set at the operating pressure desired and helps keep the circulating oil constantly heated to approximately 100°F by friction alone. If for any reason the oil cannot take its normal path, the pressure switch will start the cycle over again. The fluctuating pressure gage and flickering panel lights warn the operator when trouble occurs. One to three cycles are usually required before the oil is ready to be sprayed at any one of the 10 stations.

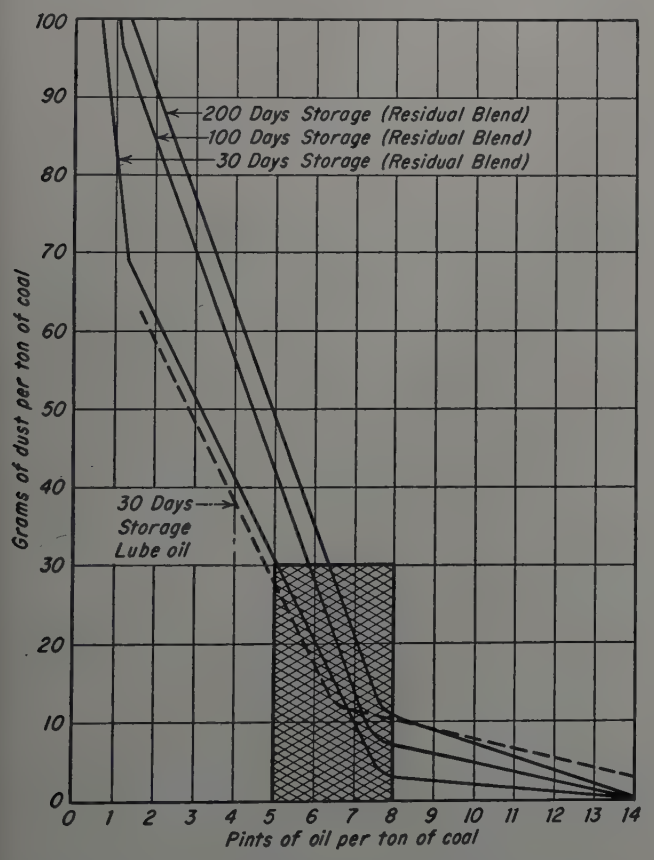


Fig. 1—Results of test made on 1¼x⅜-in. stoker coal with lubricating oils and residual blends. The average dust index for untreated 1¼x⅜-in. stoker coal is 215 g per ton of coal. The shaded area indicates where dust results are kept, including float dust.

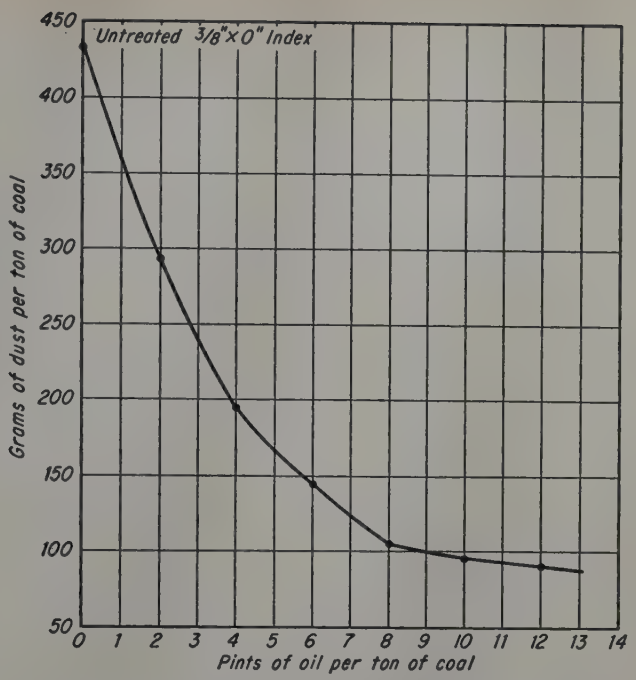
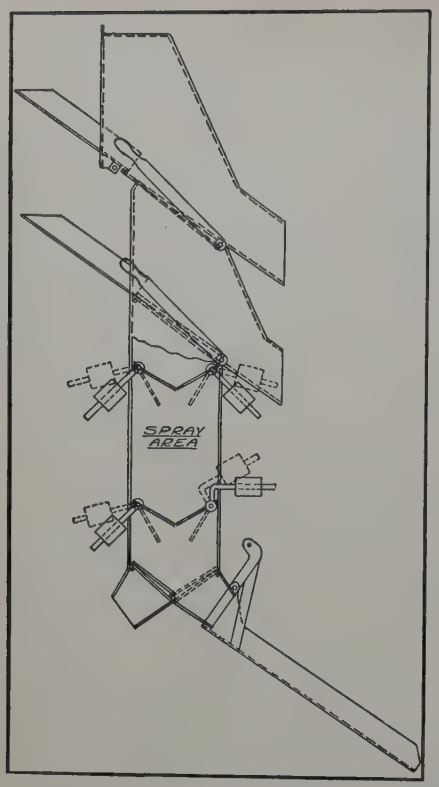


Fig. 2—Results of tests on —⅜ coal with seven oils ranging from low to high viscosity: 200 sec. pale lube oil; 1250 sec pale spray oil; 150 sec. pale spray oil; 800 sec. black spray oil; 500 sec. black spray oil; 800 sec. black spray oil; 200 sec. black spray oil. A typical screen analysis is shown in Table I.

In case of a line break with its attendant fire hazard and cleanup problem, a low-pressure switch has been installed to cut the pump motor when pressure drops below a predetermined setting. Panel lights and gage again warn the operator, who calls for a mechanic on the intercom.

High pressure meters with a flow indicator, as shown in Fig. 5, give the gallon per minute flow as the oil is being applied. Tonnage is estimated for the first car and checked and any necessary adjustments are made on the second car. Coal inspectors check meters and coal appearance at all times, recording amounts used. Solenoid valves control oil flow to any particular spraying point, but the gallon per minute flow is controlled by needle valves for each spray nozzle.

Coal sizes can be treated as follows: 1—The ⅜x0-in. coal is treated in the chamber shown in Fig. 3. 2—The 1¼x⅜-in. coal is treated in large chutes at the end of the loading boom, Fig. 3, or in a special chute carrying coal from the classifying shaker. 3—Any combination of 1½x0-in. can be handled in the large chutes shown in Fig. 3. 4—Egg sizes are handled at a transfer point on the rubber belt to prevent excessive breakage. 5—Lump coal is



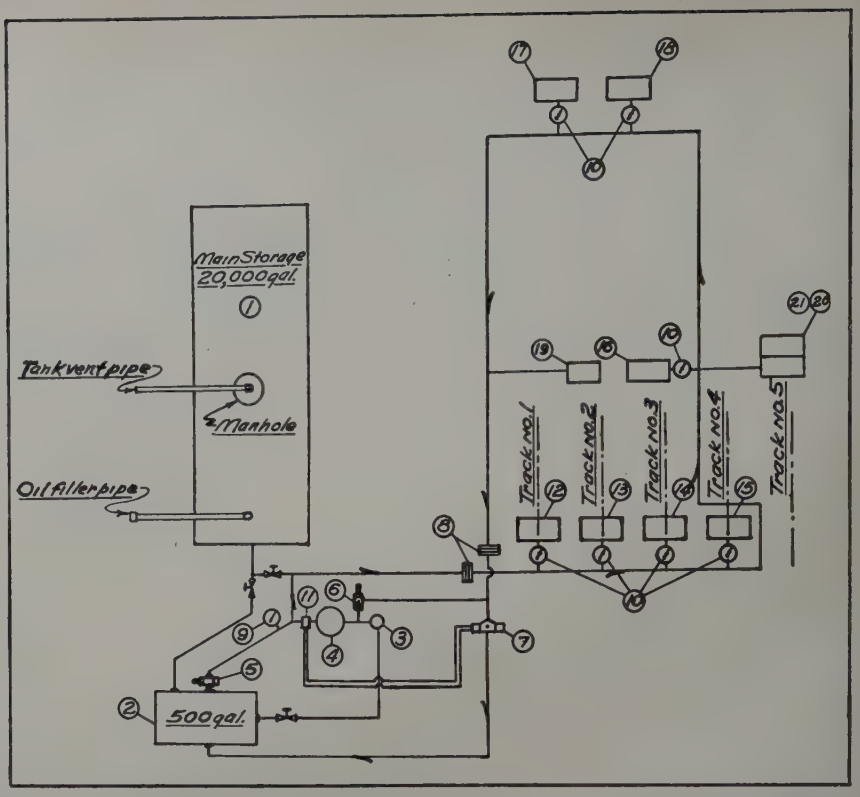


Fig. 3 (left)—The 1¼x⅜-in. coal is treated in large chutes, shown here, at the end of a loading boom, or in a special chute carrying the coal from the classifying shaker. Any combination of —1½ coal can be handled in the large chutes. Fig. 4 (right)—A diagram of the oil-treatment system at Georgetown preparation plant. Circled numbers indicate 1—the main storage tank; 2—the 500-gal heating tank; 3—the Cuno oil filter; 4—the Vickers pump (V-124), 17.5 gpm; 5—the Vickers relief valve (with spring No. 65705); 6—the Vickers relief valve (with spring No. 61470); 7—the Vickers solenoid valve; 8—insulating blocks; 9—Schraeder gage, which should read approximately 900 lb; 10—high pressure meters; 11—pressure switch; 12—spray chute, track No. 1; 13—spray chute, track No. 2; 14—spray chute, track No. 3; 15—spray chute, track No. 4; 16—egg spray, track No. 3; 17—the —⅜ slack spray chamber; 18—the 1¼x⅜-in. stoker spray chamber.

handled in an enclosed chamber over bar screens used to remove degradation and any excess oil in one operation.

To get the maximum results with a minimum of oil-fog settling out in the loading plant, the finer sizes of coal should be oil-treated by the method shown in Fig. 3. Coal is dropped through sealing flys into the spray chamber, which must be large enough to allow the coal bed to open up, exposing each individual piece to oil fog, no matter how

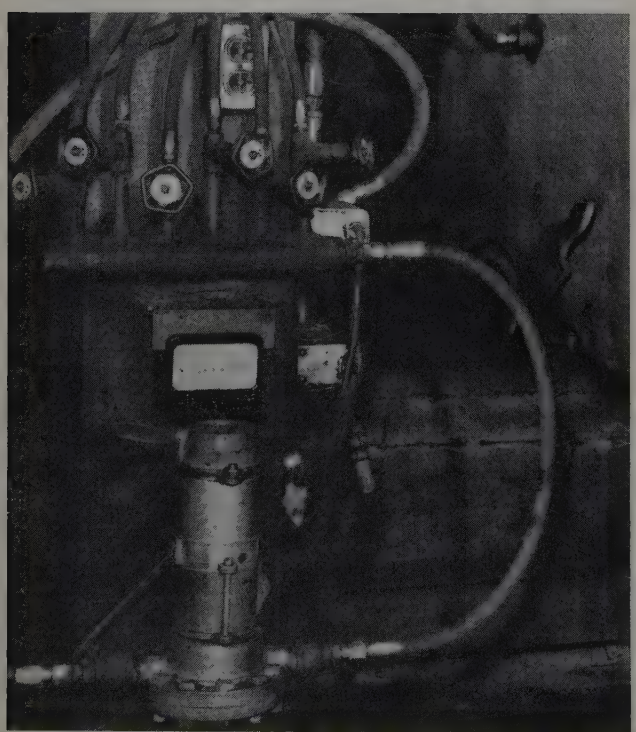


Fig. 5—High pressure meters with flow indicator give the gallon-per-minute flow.

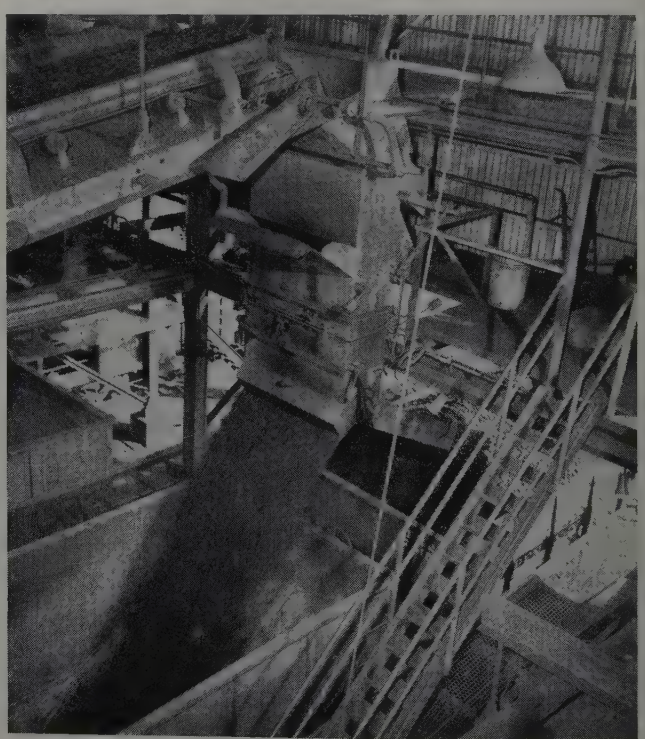


Fig. 6—A view of one of the main treating chambers being used as a loading chute.

small. The lower flys seal off the bottom and spread the coal around to pick up any excess oil that may have gathered on the sides of the chute, preventing dripping into the cars. Excellent fogging or atomizing of the oil is achieved by the higher pressures and spraying nozzles used, insuring complete coverage. Laboratory dust checks for the proper amounts of oil to be used on the coal must take into consideration the handling and breakage of the coal until it reaches the consumer. At the Georgetown preparation plant it has been found that an increase of up to one pint will handle stoker coal for the most adverse conditions. Under ideal conditions oil cannot be seen on the coal, although the product is dust-free.

Fig. 6 is a view of one of the main treating chambers being used as a loading chute. It is completely automatic and hydraulically controlled to insure proper loading of cars to a maximum tonnage with a minimum of spillage. Operation of the backloading or car-changing chute is very rapid and eliminates short shutdowns when car retarders stick, for coal can be backloaded until the car is rolling again. When a car is being loaded at a rate of 10 tons per min it is imperative that a rapid transfer arrangement be installed.

Tonnage capacity when coal is treated through the chambers is limited by coal size and type of treatment desired. At Georgetown 600 tph of 1½x0-in. coal have been treated, but above 300 tph the treatment proved to be very uneven.

Each spray chamber is equipped with sufficient nozzles to insure adequate oil for all tonnages handled. The best results seem to be with rates between 100 to 300 tph. In smaller chambers, tonnage can be dropped to a very low rate, but very small amounts should not be treated in chambers built for high tonnage, as oil fog will settle out on the side plates and drip down into the car. The area for spraying must be large enough to permit easy coal flow and yet small enough to prevent oil fog from gathering and dripping.

Pipe Resistance Method

It was originally thought that for proper treatment heavier oils were necessary, and resistance heating was incorporated so that any viscosity could be handled with equal success. It is now known that it is not necessary to go above 300-sec oil and so the heating system is a standby unit at present. If use of heavier oils were continued the following would hold true.

Constant temperature, as well as pressure, is important to insure an even flow of oil through the small orifices in fogging or spray nozzles. Forcing oil through a spool-type relief valve at 17½ gpm keeps the temperature about 100° to 120°F, depending on weather conditions. Auxiliary heat to raise this to 150°F or more is needed if the higher (1000+) viscosity oils are to be used successfully.

Welding machines of 300 amp can be used as the heat source. Special electrical insulators were designed for the 1-in. line to hold the high pressures and still be flexible enough to seal after alternate heating and cooling with accompanying expansion and contraction. Wherever a hydraulic hose was inserted, a jumper of copper wire was installed to maintain the circuit without the danger of overheating caused by a poor connection within the hose. Line resistance does not actually heat the oil passing through the circuit; it heats the conductor, which in turn keeps the oil next to the pipe in a fluid condition during extremely cold weather. Resistance heating keeps stationary oil in a fluid state and offsets heat loss due to radiation in a circulating system. In actual practice oil of 800 to 1000 viscosity has been forced through cold lines without trouble because of the precautions and valving arrangements. It is recommended that for greater viscosity additional heating facilities such as the pipe resistance method be used to insure movement of oil in the circuit under adverse weather conditions.

Tests have proved that a light, pale, odorless oil best satisfies customer demands for the particular coal treated at Georgetown. The spraying system is flexible enough to handle almost any oil, making it possible for the company to take advantage of price drops in distress oils, when the product is compatible with stocks on hand. In the past two years, various oils have been sprayed at Georgetown, ranging from residuals to lubricating stocks and varying in viscosity from 200 to 2000 at 100°F.

The following results have been taken from reports of coal shipments for the lake trade, where coal is handled in great amounts under all weather conditions.

1—The product is dust free. 2—Less moisture is absorbed when cars are caught in bad weather, and faster dewatering is possible. It follows that 3—coal is easier to move in cold weather, and when coal is frozen, the oil film prevents particles of coal from freezing solidly to the steel cars or to each other. To carry this advantage further, spraying equipment is being installed to treat empty railroad car sides and slopes prior to loading. 4—Treated coal moves more easily in cars, in stockpiles, or on conveying equipment.

The task of treating coal properly is by no means a hit-and-miss proposition. Suitable oils and methods must be used and should be made foolproof by constant quality control checks of the oil when it is delivered and the coal as it is loaded. There are no shortcuts to proper treatment.

Corrections

In the November 1953 issue: TP 3556A. The Status of Testing Strength of Rocks. By Rudolph G. Wuerker. P. 1113, Acknowledgments, should read as follows: This work is part of a comprehensive program of investigating strength properties of rocks undertaken by the Department of Mining and Metallurgical Engineering at the University of Illinois. The wholehearted support given the project by Professor H. L. Walker, Head of the Department, is herewith gratefully acknowledged. The tests performed at the University were made in the Department of Theoretical and Applied Mechanics.

In the December 1953 issue: TP 3642L. The Scintillation Counter in the Search for Oil. By R. W. Pringle, K. I. Roulston, G. M. Brownell, and H. T. F. Lundberg. P. 1257, captions for Figs. 3 and 4 should be transposed to read as follows: Fig. 3—Resolution energy relationship. Fig. 4—Th gamma ray scintillation spectrum. In the list of authors, p. 1255, G. M. Brownell's name should read as given here.

Tumbling Mill Capacity and Power Consumption as Related to Mill Speed

by R. T. Hukki

THE accepted basis of comparisons between mills of different diameter is the percentage critical speed.

If n = actual mill speed, rpm,
n_c = calculated critical speed, rpm,
n_p = calculated percentage critical speed, and
D = inside diameter of the mill in feet,

$$\text{then } n_c = 76.63 \frac{1}{\sqrt{D}} \text{ rpm} \quad [1]$$

$$n_p = \frac{n}{n_c} 100 \text{ pct} \quad [2]$$

$$n = 0.7663\, n_p \frac{1}{\sqrt{D}} \text{ rpm.} \quad [3]$$

In the following analysis capacity, T, is expressed in short tons per hour, tph, and power consumption, P, in kilowatts, kw. Accordingly power consumption per unit of capacity, P_o, will be expressed in kilowatt hours per short ton, or kw-hr per ton. In all equations D refers to the inside diameter of the mill in feet and v to the peripheral speed of the mill in feet per minute inside the liners. Comparison between separate mills must be based on equivalent grinding conditions, i.e., same feed, same size distribution of feed, same size distribution of product, and same percentage of solids. In addition, comparisons between separate rod mills must be based on the same rods, same type of liners, and same percentage rod load. Comparisons between separate ball mills presuppose the same balls, similar liners, and same relative ball load. The practical n_p-range through which the equations apply varies, being narrower for fine grinding in ball mills and wider for coarse crushing in rod mills.

The Relationship between Capacity and Speed

It is the general belief that the capacity, T, of a tumbling mill is directly proportional to the speed of the mill, other things remaining constant.[1] Mathematically this is represented by the equation

$$T = c_1 n \text{ tph} \quad [4]$$

where c_1 is a factor related with the grinding characteristics of the ore, method of reduction, and the units chosen.

It is proposed here that the general equation relating mill capacity and speed should be of the form

$$T = c_1 n^m \text{ tph} \quad [5]$$

In other words, the capacity should be proportional to the mill speed raised to power m, the numerical value of the exponent being $1 \leqq m \leqq 1.5$, depending on the circumstances. Eq. 5 can also be written in the following forms:

$$T = c_2 (n_p)^m \text{ tph, and} \quad [6]$$

$$T = c_3 v^m \text{ tph,} \quad [7]$$

where v = peripheral speed of the mill in feet per minute.

If the observed capacity of a mill at speed n_1 is = T_1 tph, the capacity T_2 of the same mill at speed n_2 should be

$$T_2 = T_1 (n_2/n_1)^m \text{ tph} \quad [8]$$

The Relationship between Power Consumption, Mill Diameter, and Speed

The only well known theoretical deduction relating power consumption, P, and mill diameter appears to be the formula of duPont introduced by Gow, Guggenheim, Campbell, and Coghill.[2] According to duPont, the power required to operate a mill is a function of the mass of the balls, of the lever arm of the ball mass, and of the speed of the mill. The ball mass per unit of mill length is proportional to the square of the diameter, the lever arm is directly proportional to the diameter, and the critical mill speed or any percentage thereof is inversely proportional to the square root of the mill diameter. Following this reasoning, the original duPont formula is of the form

$$P = c_4 D^2 \cdot c_5 D \cdot c_6 D^{-0.5} = c_7 D^{2.5} \quad [9]$$

If the mill speed in the above equation is expressed in terms of Eq. 3, the duPont formula may be written as follows:

$$P = f_1(D^2) \cdot f_2(D) \cdot f_3\left(\frac{n_p}{\sqrt{D}}\right), \text{ or} \quad [10]$$

$$P = c_8 n_p D^{2.5} \text{ kw} \quad [11]$$

Eq. 11 may also be derived from the mechanical principle of force, which is equal to mass × acceleration. Power necessary to operate a mill may be considered to be an homogeneous linear function of the force developed. Ball or rod mass per unit of mill length is a function of D^2. The acceleration factor of the ball or rod mass is a function of the peripheral speed of the mill. Thus

$$P = f_4(F) = f_1(D^2) \cdot f_5(v).$$

Indicating that $v = \pi D n$, and $n = c_9 n_p/\sqrt{D}$, the above equation becomes $P = f_1(D^2) \cdot f_5(\pi D\, c_9\, n_p/\sqrt{D}) = c_8\, n_p\, D^{2.5}$.

R. T. HUKKI, Member AIME, is Professor of Mineral Dressing, Finland Institute of Technology, Helsinki, Finland.

Discussion on this paper, TP 3833B, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Jan. 18, 1954.

Power consumption according to Eq. 11 must be considered as a first approximation only because the formula does not take into account the effect of mechanical losses such as friction in the bearings and gears.

The Relationship between Capacity, Mill Diameter, and Speed

As a first approximation, the capacity, T, of a mill may be considered as a function of the force acting inside the mill. Therefore

$$T = f_6(F) = f_1(D^2) \cdot f_7(v) \text{ tph} \qquad [12]$$

It was proposed earlier that capacity of a mill should be proportional to the peripheral speed raised to power m (Eq. 7). Correspondingly, the corrected formula for capacity should be of the following form

$$T = f_1(D^2) \cdot f_7(v^m) \text{ tph} \qquad [13]$$

Indicating again that $v = \pi D n$, and $n = c_9 n_p / \sqrt{D}$, Eq. 13 becomes

$$T = c_{10}(n_p)^m D^{(2+m/2)} \text{ tph} \qquad [14]$$

which is the proposed basic equation relating capacity, mill diameter, and speed. If $m = 1.0$, Eq. 14 becomes

$$T = c_{10}\, n_p\, D^{2.5} \qquad [15]$$

If $m = 1.5$, it becomes

$$T = c_{10}(n_p)^{1.5} D^{2.75} \qquad [16]$$

The Relationship between Power Consumption per Ton, Mill Diameter, and Speed

The power (P_o) required for reduction of a unit weight of rock is equal to the total power consumed (Eq. 11) divided by the total tonnage handled (Eq. 14).

$$P_o = P/T = \frac{c_8\, n_p\, D^{2.5}}{c_{10}(n_p)^m D^{(2+m/2)}} \text{ kw-hr per ton}$$

from which

$$P_o = c_{11}(n_p)^{(1-m)} D^{(0.5-m/2)} \text{ kw-hr per ton} \qquad [17]$$

It can be seen from Eq. 17 that for $m = 1.0$, P_o is independent of the mill diameter and mill speed. Should m be > 1.0, the value of P_o decreases with increasing values of D and n_p. Should the value of $m = 1.5$, P_o decreases in proportion to $1/\sqrt{n_p}$, if the mill diameter remains constant, and P_o decreases in proportion to $1/\sqrt[4]{D}$, if the percentage critical speed of the mills under comparison remains the same.

Again, Eq. 17 gives a first approximation only. If the power drawn by the motor is the measured quantity as is the usual case, it will be consumed by mechanical losses, heat developed, and useful reduction work done. Eq. 17 will be correct only upon the condition that proportional distribution of energy between the three major components remains constant.

Discussion

The cornerstone of the mathematical treatment presented is the relationship between mill capacity and speed as expressed in Eq. 5. To prove it, the writer can introduce only one series of experimental evidence. This series is the result of the outstanding rod mill test performed by Banks in the Sullivan concentrator.[8] Respective numerical data are given in Table I. As will be seen from Fig. 1, the data give a straight line on logarithmic paper for the n_p-range of 50 to 85 pct. The slope m of the line is very close to 1.5.

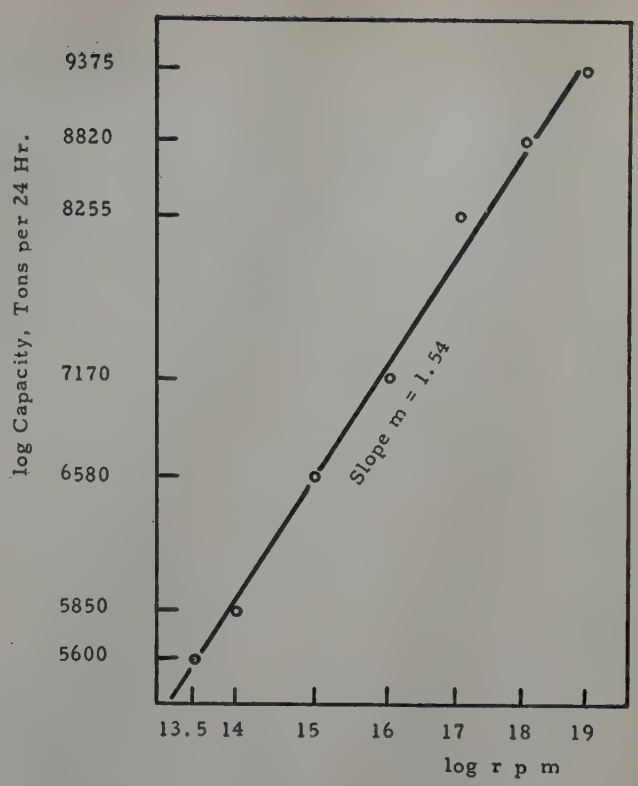


Fig. 1—Graphical presentation on a logarithmic paper of the results of the rod mill test by Banks.

Table I. Rod Mill Data by Banks

Speed, rpm	13.5	14	15	16	17	18	19
Tons per 24 hr	5600	5850	6580	7170	8255	8820	9375

The Sullivan rod mill with its fluid drive coupling has made this test possible, a test out of the reach of practically all other grinding installations. This may be why the relationship between mill capacity and speed has not been studied earlier in more detail and why the knowledge of this relationship has been based until now on crude observations.

The result of the Sullivan test, although startling, seems to have sound fundamental reasons. Considering the principle of force = mass × acceleration, the acceleration factor of falling rods increases more rapidly than peripheral speed with increasing mill speed. The application of greater force at higher speed will increase the probability of effective reduction, especially of the coarser fraction of feed.

The situation demonstrated in the Sullivan concentrator can undoubtedly be duplicated in other coarse-crushing rod mills. The slope of the line may or may not be equally steep in controlling rod mills, using the terminology introduced by Myers.[4] Slopes greater than 1.0 should be anticipated for ball mills grinding coarse ore. At present it is difficult to find evidence whether or not the numerical value of the slope would diminish toward 1.0 with ball mills grinding successively finer and finer feeds.

Extensive investigations have been undertaken to study the relationship between the power and capacity and the mill diameter of ball mills. As a result, both power and capacity have been expressed

as a function of mill diameter D raised to power p. The numerical value of exponent p was found to be 2.6 by Gow, Campbell, and Coghill,[5] 2.65 by Fahrenwald and Reck[6] for laboratory mills, 2.6 by the Lake Shore staff[7] for commercial ball mills, and for the capacity a variable figure from 2.5 for fine grinding in low-speed mills to 3.0 for coarse grinding in high-speed mills by Bond.[1] It should be emphasized that in none of these evaluations has the parameter of mill speed been included in the formula, although Bond seems to appreciate its effect by giving different values of the exponent p for mills of low and high speed.

The equations introduced in this paper seem reasonable in the light of the evidence cited above. The proposed equation, Eq. 14, for mill capacity agrees well with Bond's principle of varying exponent. Unfortunately the writer has not been able to find published data to check his equations against the respective relationships by Bond, who writes as follows:[1] "It is generally, but not universally, held that large-diameter mills have a higher mechanical efficiency than small mills or, in other words, that the capacity increases at a higher exponent of the diameter than 2.5, resulting in lower kwh/ton value for the larger mills."

Eqs. 11, 14, and 17 satisfy the requirements of this statement and seem to be in good agreement with the present-day trend toward very large diameter mills in grinding.

On the basis of the equations developed the following relationships should exist:

1—If the speed of a coarse-crushing rod mill is increased from n_p-value of 50 pct to n_p-value of 80 pct, its capacity will be doubled.

2—If the mill diameter is doubled and n_p remains constant, the increase of the mill capacity is 5.6 or 6.7-fold, depending on the value of the exponent of D ($p = 2.5$ or 2.75, respectively).

3—If the diameter of a coarse-crushing rod mill is doubled and the n_p-value increased at the same time as indicated in point 1, its capacity will be 13.4 times the original.

4—If the liner wear of a 6-in. mill is 2 in., the mill diameter will be increased by wear from 6 in. up to 6 ft 4 in. The effect of the increasing diameter on mill capacity will be 14.4 or 16.0 pct, depending on the exponent of D. At the same time, while the revolution per minute value of the mill remains constant, the n_p-value increases 2.8 pct. The minimum value of the final capacity will therefore be $= (102.8/100) \times 114.4 = 117.6$ pct and the maximum value $= (102.8/100)^{1.5} \times 116.0 = 120.9$ pct of the original, or the increase of the capacity 17.6 and 20.9 pct, respectively.

5—If the diameter of a coarse-crushing rod mill is doubled and the n_p-value remains constant, the power consumption per ton should decrease 15.9 pct.

The analysis presented above deals with the fundamentals of grinding. It is not possible for the writer to prove or disprove by experiment. Therefore the paper is presented as an invitation for discussion to the mill men in general. Their judgment, whatever it may be, would advance the present knowledge of the science of grinding.

References

[1] F. C. Bond: Mathematics of Crushing and Grinding. *Recent Developments in Mineral Dressing*, pp. 101-115. The Institution of Mining and Metallurgy, London (1953).

[2] A. M. Gow, M. Guggenheim, A. B. Campbell, and W. H. Coghill: Ball Milling. *Trans.* AIME (1934) **112**, pp. 24-78.

[3] H. R. Banks: The Rod Mill in the Sullivan Flow. *Trans.* CIMM (1952) **55**, pp. 255-261.

[4] J. F. Myers: Defining the Scope of the Open Circuit Rod-Mill in Comminution. *Recent Developments in Mineral Dressing.* The Institution of Mining and Metallurgy, London (1953) pp. 137-150.

[5] A. M. Gow, A. B. Campbell, and W. H. Coghill: A Laboratory Investigation of Ball Milling. *Trans.* AIME (1930) pp. 51-81.

[6] A. W. Fahrenwald: Some Fine-grinding Fundamentals. *Trans.* AIME (1934) **112**, pp. 88-115.

[7] Staff, Lake Shore Mines: Fine Grinding Investigations at Lake Shore. *Trans.* CIMM (1940) **43**, pp. 299-434.

Activation Energies for the Decomposition Of Limestone, Dolomitic Limestone, and Dolomite

by J. H. Wernick

IN a study of the rate of decomposition of ½-in. cubes of limestone, dolomitic limestone, and dolomite in a sweeping nitrogen atmosphere, Joseph, Beatty, and Bitsianes[1] found that the zone of calcination advanced at substantially a constant rate at a given temperature. The purpose of this paper is to show that their data indicate at least two processes occurring when the above carbonates decompose. It is suggested that there are two processes occurring consecutively, one the limiting process in a given temperature range and the other the limiting process over another temperature range.

J. H. WERNICK, Junior Member AIME, is Instructor in Metallurgy, The Pennsylvania State University, State College, Pa.

Discussion on this paper, TP 3834H, may be sent (2 copies) to AIME before Sept. 30, 1954. Manuscript, Oct. 6, 1953.

The data of Joseph and co-workers have been extended by use of the Arrhenius equation, Eq. 1, a relation beween the velocity constant k of a reaction and the absolute temperature T.

$$\frac{d \ln k}{dT} = \frac{A}{RT^2} \qquad [1]$$

A is the activation energy in calories per mol (additional energy that must be added for an average molecule to react), and R is equal to 1.986 cal

per mol per degree. The integrated form of this equation is

$$\ln k = -\frac{A}{RT^2} + C \qquad [2]$$

where C is the constant of integration. This is the equation of a straight line. If ln k is plotted against $\frac{1}{T}$, the slope of the line is equal to $-\frac{A}{R}$ and the activation energy, A, can be calculated.

The specific rate data measured by these workers are plotted against $\frac{1}{T}$ in Fig. 1. The full circles are the experimentally determined points and the open circles are points taken from the curves drawn through their experimental points. The open circles were used to determine more closely the slopes of the high and low temperature portions of the smooth curves. The plots show a gradual change in the slopes of the low-temperature portion of the curves as the temperature is increased, indicating a change in the limiting process. This writer's activation energies A_1 and A_2, corresponding to the low and high-temperature portion of the curves respectively, are given in Table I.

At low temperatures the reaction having the larger A value (A_1) is the observed rate-controlling process, and at the high temperatures the reaction having the smaller A value (A_2) dominates. The A_1 values are of the same order of magnitude as the heat of dissociation of $CaCO_3$ at 800°C, namely 40,000 cal per mol, and it appears that activation energy for the reverse process will be small, so the state consisting of CaO and free CO_2 gas lies close to the state corresponding to the crest of the energy barrier (state of the activated complex). This quantitatively accounts for the observation that CaO picks up CO_2 readily from the atmosphere. It is interesting to note that the A_1 values are of the same order of magnitude as the resonance energy (42,000 cal per mol) for the carbonate ions in the dialkyl carbonates for resonance of the double bond among three positions.[2] The resonance energy is the additional stabilizing energy of the carbonate ion, and at least this amount of energy must be added to an average molecule before decomposition can occur.

Table I. Activation Energies for the Decomposition of Dolomite, Dolomitic Limestone, and High Calcium Limestone

Item	A_1 (Cal Per Mol)	A_2 (Cal Per Mol)
Dolomite	43,250	19,620
Dolomitic limestone	46,000	19,900
High calcium limestone	46,000	25,800

The region of temperature where there is a gradual change in the slope of the low temperature portion of the curves as the temperature increases can be attributed to a situation where both processes are occurring at comparable rates and neither predominates. As a result of this the observed rate at a given temperature within this temperature region will be determined by the rate at which both processes occur. In other words, the differential equation expressing the rate process at a given temperature will contain the specific rate constants of both consecutive processes occurring, and none can be neglected. In this temperature region, the effect of

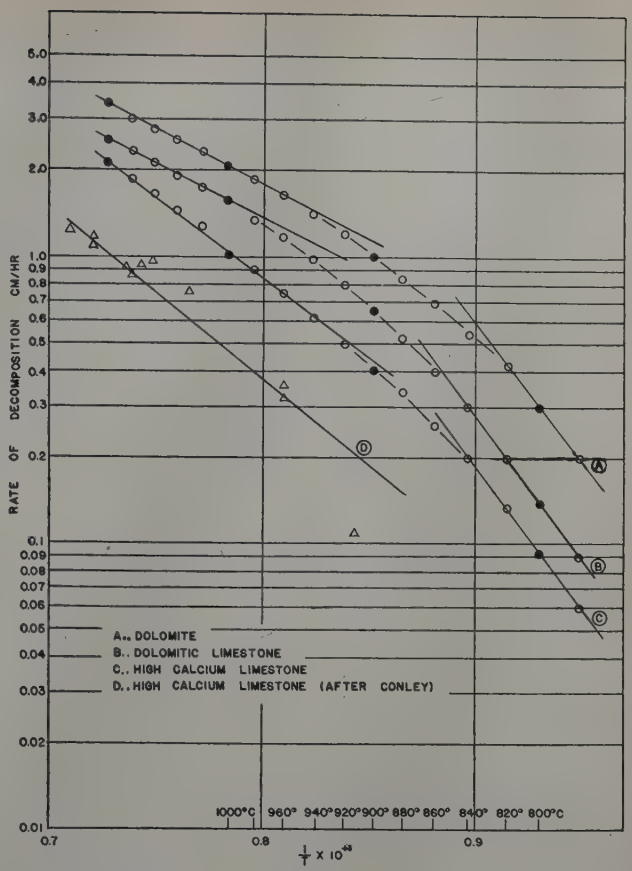

RATE OF DECOMPOSITION GM/HR
A.. DOLOMITE
B.. DOLOMITIC LIMESTONE
C.. HIGH CALCIUM LIMESTONE
D.. HIGH CALCIUM LIMESTONE (AFTER CONLEY)
1000°C 960° 940° 920° 900° 880° 860° 840° 820° 800°C
1/T x 10^4

Fig. 1—Rate of decomposition as a function of temperature.

temperature will be complex, since both processes will have different temperature coefficients. At sufficiently low temperatures or at sufficiently high temperatures, where one of the two processes occurs so slowly in comparison to the other, the observed rate is determined essentially by the slowest process and the observed temperature effect will be given by the Arrhenius equation.

The justification for splitting the curves of Joseph et al into two straight lines, even though there are only four experimental points, is derived from other experimental work. For example, Bischoff[3] studied the decomposition rate of dolomite and limestone in various atmospheres in the temperature range 550° to 800°C. For dolomite, he found the A values to be 44,238 cal per mol for dry air, 40,939 cal per mol for moist air, and 26,670 cal per mol for steam. For limestone, in dry air and steam, he calculated A values of 47,664 and 38,972 cal per mol respectively. The reactivity of carbonates is greatly influenced by water vapor.[4] These A values substantiate the A_1 values of Joseph and co-workers if the steam data are excluded. Conley[5] determined the rate of decomposition of a high calcium limestone at temperatures above 916°C under one atmosphere of CO_2 (equilibrium decomposition temperature approximately 900°C.) His data are plotted also on Fig. 1, line D. The line drawn through the experimental points has the same absolute slope as the line drawn by Conley. From his rate data, this writer has computed an activation energy of 27,000 cal per mol, in agreement with the A_2 value of Joseph and coworkers for the same material. The high activation energy, A_1, at the lower temperatures, may be due to the probably difficult process of removing CO_2 molecules from the $CO_3^=$ ions in the hexagonal lattices of calcite and dolomite. The moment decomposition

starts in $CaCO_3$, for example, Ca^{++} ions and the remaining $O^{=}$ ions must, by diffusion, form the cubic CaO lattice. It is not until the higher temperatures are reached that this latter diffusion process becomes sufficiently dominant, giving rise to a lower activation energy. Barrer[6] points out that the activation energy for diffusion in ionic crystals is smaller than the activation energy for diffusion in metals and that this is due to the lower densities of salts and the greater effect of polarization forces. However, this comparison is based on data for diffusion in ionic crystals where the cation or anion or both are univalent. Actually data for diffusion in bivalent ionic crystals of the RX type are insufficient to permit positive naming of the diffusion process as the limiting one at the elevated temperatures. For the reverse process (combination of CaO with CO_2) diffusion of Ca^{++} and $O^{=}$ ions will be the limiting process at the lower temperatures.

The effect of water vapor in increasing the rate of decomposition of limestone and dolomite (lowering of the activation energy) mentioned above can possibly be explained on the basis of the dipole nature of a water molecule[2] (unsymmetrical charged entity for practical purposes.) Conceivably the dipoles can interact with the bonding situation at the reacting interface to weaken the structure and permit CO_2 to escape more readily. Such types of interactions might also be used to explain some of the other results of Bischoff[7] and the fact that H_2 readily reacts with limestone at 600°C.[8] In the latter case, H_2 enters into chemical combination with the CO_2 to produce CO and H_2O.

The rate data of Ralston, Pike, and Duschak[9] for the decomposition of magnesite under one atmosphere of CO_2 in the temperature range of 600° to 850°C indicate that a change in the controlling reaction takes place at about 675°C. If the logarithm of the time for 50 pct decomposition is taken from their data and plotted against $1/T$, the A_1 values vary between 24,400 and 32,000 cal per mol, and the A_2 values are about 17,000 cal per mol. Their low temperature data, however, are meager and the A_1 values were calculated on the basis of only two points. From heat of formation data at 20°C and heat capacity data, the heat of decomposition of $MgCO_3$ was calculated to be about 25,100 cal per mol at 627°C and is of the same order of magnitude as the above A_1 values. Bischoff[7] determined an activation energy of 36,128 cal per mol for decomposition of $MgCO_3$ in vacuo in the temperature range 400° to 600°C. The smaller A_1 value for magnesite compared to calcite may be attributed to a greater polarizing effect by the $CO_3^{=}$ ion on the smaller Mg^{++} ion and consequently, a stronger bond between the Mg^{++} and $CO_3^{=}$. As a result of this, the $CO_3^{=}$ ion would be weaker in so far as removal of CO_2 is concerned. The smaller Mg^{++} ion (radius of 0.72A)*

* Average of values of Pauling and Goldschmidt.[2]

might also account for the lower A_2 values of Ralston and co-workers for magnesite. The idea of the greater ease with which the smaller ion may diffuse, giving rise to a smaller activation energy, is supported by the lower A_2 values derived from the data of Joseph and co-workers for the dolomite and dolomitic limestone.

Spencer and Topley[4] studied the rate of decomposition of chemically prepared crystalline Ag_2CO_3 in vacuo in the temperature range 147° to 225°C, and for three different preparations, A, B, and C, they calculated activation energy values equal to 23,400, 22,800, and 22,500 cal per mol respectively. They showed that the ratio of the linear rate of propagation of the reaction to the initial radius of the particles was substantially a constant during the course of the reaction at constant temperature. The heat of dissociation of Ag_2CO_3 at 20°C, calculated from heat of formation data, is about 19,550 cal per mol. Because of the lack of sufficient heat capacity data, the heat of the reaction at higher temperatures cannot be calculated, but it will be somewhat less at the temperatures at which Spencer and Topley worked. Presumably this range of temperatures corresponds to activation energy values which may be labeled as A_1, and for this carbonate also the final state lies close to the state corresponding to the crest of the energy barrier.

Conclusions

It appears that there is a change in the rate-controlling process for the decomposition of limestone, dolomitic limestone, and dolomite as the temperature is raised. It is suggested that the breakup of the carbonate ion is the limiting process at temperatures below about 880°C, the diffusion of the cations and $O^{=}$ ions being the rate-determining process above this temperature.

It is believed that since the various carbonate compounds encountered in nature decompose readily at different temperatures, a fruitful line of reasoning and experimentation to explain this fact can be based on fundamental differences of compounds.

It is clear that the atmosphere in which the carbonates are decomposing can influence decomposition rate. It is suggested in this paper that this effect might be attributed to fundamental interactions between the bonding situation at the surface of carbonates and molecules of the gaseous atmosphere.

Acknowledgment

The writer wishes to thank H. M. Davis, R. W. Lindsay, J. W. Fredrickson, A. J. Shaler, and the other members of the staff of the Division of Metallurgy, The Pennsylvania State University, for their helpful discussions during the preparation of this paper.

References

[1] T. L. Joseph, H. M. Beatty, and G. Bitsianes: Calcination Rates and Sizing of Blast-Furnace Flux. *Trans.* AIME, **154**, pp. 148-161.

[2] L. Pauling: *Nature of the Chemical Bond*, p. 208. Cornell University Press, 1945.

[3] F. Bischoff: Kinetics of Thermal Dissociation of Dolomite and Limestone in Various Atmospheres. *Ztsch. anorg. allgem. Chemie* (1950) pp. 262, 288-296.

[4] W. D. Spencer and Bryan Topley: Chemical Kinetics of the System $Ag_2CO_3 = Ag_2O + CO_2$. *Journal of the Chemical Society* (London, 1929) pp. 2633-2650.

[5] J. E. Conley: Calcination Conditions for Limestone, Dolomite, and Magnesite. *Mining Technology* (March 1939).

[6] R. M. Barrer: *Diffusion In and Through Solids*. Cambridge University Press, 1941.

[7] F. Bischoff: On the Process of Magnesite Calcination in Air, in Vacuum, and in Various Foreign Gases. *Radex-Rundschau* (May 1950) pp. 141-147.

[8] G. Bitsianes, J. H. Wernick, and T. L. Joseph: Unpublished work, University of Minnesota.

[9] O. C. Ralston, R. D. Pike, and L. H. Duschak: Plastic Magnesia. U. S. Bur. Mines *Bull. 236* (1925).

A Milestone in Subsurface Exploration

The NX Borehole Camera

by E. B. Burwell, Jr., and R. H. Nesbitt

Designed by army engineers, the NX borehole camera provides the most economical method of subsurface exploration so far devised. Continuous cylindrical color photographs now reveal minor flaws in foundation bedrock not always disclosed by borehole samples. A unique projector transforms the photographs into true-to-scale images of three dimensions.

A NEW milestone in the progress of photography and subsurface exploration was reached when the Chief of Army Engineers announced the development of the NX borehole camera. In obtaining continuous undistorted cylindrical color pictures of dry or water-filled borings the device surpasses all other cameras designed for photographing interior surfaces of pipes, wells, and conduits.

The usefulness of the camera and the unique projector that comes with it are at once apparent. To the geological, mining, and civil engineer alike it signifies the end of guesswork with respect to underground conditions that have eluded identification by conventional core-drilling methods. To the medical profession it may well signal new progress in photo optics which could result in cylindrical color photographs supplementing the X-ray and existing photographic methods used in exploratory surgery.

The pilot model of the camera has more than paid for itself as a result of its success in bringing to the surface at two dams information previously obtainable only by costly drilling of mansized boreholes for subsurface on-the-spot examination. Such bores cost as much as $200 per lineal foot as against $10 per ft for the conventional 3-in. or NX exploratory core borings from which the present invention derives its name. The NX camera can now photograph completely the interior surface of these borings.

Research and development for the first camera and projection equipment were conducted by Engineering Research Associates, Division of Remington Rand, Inc., under the technical supervision of the Geology and Geophysics Branch, Office of the Chief of Army Engineers in Washington, in accordance with plans and specifications furnished by this branch. Subsequent modifications of the camera-lowering device, transporting equipment, and special field tools were made in the Corps of Engineers' Ohio River Division Laboratories in Cincinnati. The background of this development is interesting.

Among the more difficult foundation problems involved in construction of concrete dams are those resulting from bedrock imperfections that escape disclosure by conventional exploration methods. Often these imperfections are of sufficient magnitude to introduce costly changes, especially when their presence is discovered only after foundation excavation is well in progress. Bedrock flaws most frequently camouflaged may be ruptures resulting from the enormous stresses to which rocks have been subjected, or they may be planes or zones of weakness related to chemical alteration and underground erosion. Obviously, detailed information on their location, dimensions, and structure is important, as these factors can affect the stability of a dam and the security of life and property downstream. Their discovery and appraisal entail extensive subsurface exploration.

At present the most economical exploratory tool available to geologists and engineers is the small-diameter diamond core drill by means of which cores of the foundation bedrock are recovered for examination and testing. All too often, however, these samples fail to disclose minor foundation flaws that may be of major importance before a structure is completed. As a result, the small-diameter drillings are supplemented generally by more reliable but much more costly shafts, tunnels, or large-diameter calyx drill holes that permit the investigator to examine the foundation rocks in place. It was to reduce the number and cost of these expensive and time-consuming investigations, as well as to obtain more complete information on rock structure from the smaller borings, that the NX borehole camera was developed. That it has been eminently satisfactory in accomplishing that purpose is demonstrated by its success most recently at the Folsom dam, under construction on the American River, 23

E. B. BURWELL, JR., and R. H. NESBITT are with the Department of the Army, Office of the Chief of Engineers, Washington, D. C.

Discussion on this paper, TP 3831A1, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, March 10, 1954. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 732. The intervening non-Transactions pages appeared in Mining Engineering.

Figs. 1a-1d (left to right): 1a—Cylindrical projection of picture image shown in Fig. 1b. Note peak of conical prism which refracts plane images to three-dimension pictures on the cylindrical screen. Fig. 1b—Similar to Fig. 1d. Shows plane image projection of a faulted and fractured section of granite at a shallower depth in the same boring. Fig. 1c—Cylindrical projection of the picture image shown in Fig. 1d. Approximately one-half natural scale. Fig. 1d—Borehole camera picture of a blue-gray granite in central California. Picture, with the open center containing a compass arrow, represents a 360° conical image of the interior surface of a 3-in. boring. Approximately one-half natural scale.

miles upstream from Sacramento, Calif. Here some seventeen 3-in. boreholes, drilled to penetrate a fault zone in the granite at depths of 50 to 75 ft below the foundation, were completely photographed in kodachrome. Representative black and white reproductions of these kodachromes are shown in Figs. 1a to 1d. In addition to locating and photographing the fault zone precisely, the pictures recorded delicate changes in rock coloring, fractures as small as 1/100 in., and the surface of the groundwater table. A compass, visible in each picture, see Figs. 1b and 1d, permitted orientation of all these features on a unique cylindrical projection screen. Since the materials in the fault zone were too soft to be recovered completely by the small-diameter core drill, their investigation and the extent of the fault as a whole could be accomplished only by tunnels, shafts, and costly mansized calyx borings. The camera came to the rescue in time to eliminate the proposed calyx borings by producing faithful color-picture records of all 17 small-diameter holes. Previously the camera had been used successfully in locating cracks in the spillway of a high concrete dam and in photographing bedrock texture and structure in dam foundations in Virginia and Pennsylvania.

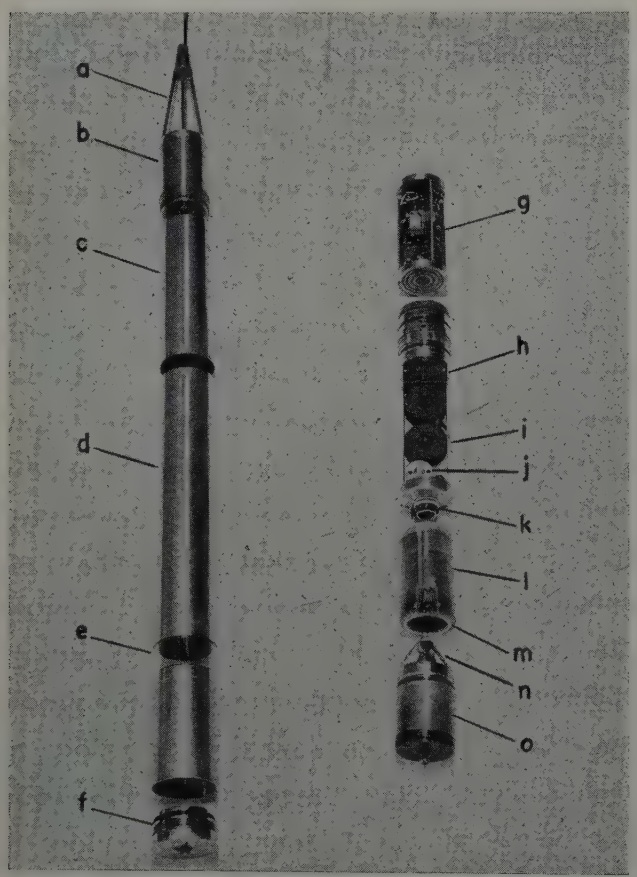


Fig. 2—A view of the NX borehole camera showing external elements of camera tubing on left, internal elements on right. (Tapering of lefthand figure is due to off-center position of photographer's camera.) Reference letters are covered in the text.

Externally the camera unit, shown in Fig. 2, is a simple stainless-steel tube, length 31½ in. and diam 2¾ in., with a circular quartz window *(e)*, not unlike a miniature lighthouse window, located 5 in. above the lower end. Internally the principal elements include an oil-damped compass *(o)* which supports a hollow and truncated conical mirror *(n)*. The mirror is situated inside and directly opposite the quartz window and thus comprises the eye of the camera. The hollow and truncated construction of the conical mirror makes the underlying compass visible to the 15-mm camera lens *(k)* located a short distance above the mirror. A high-voltage circular flash tube *(m)*, midway between the cone mirror and camera lens and actuated by a current pulsing device on the camera lowering rig, simultaneously illuminates the boring and exposes the film to the bright mirror image. Directly above the lens is a conventional 16-mm motion picture camera *(h, i, j)*, with motor and spool drive synchronized by the same pulsing circuit which actuates the flash tube. A power condenser and relay unit *(g)* contacts the top of the camera unit. The camera is designed to make 16 flash exposures per ft of boring. Thus with a 1-in. section of drillhole exposed at each flash and with 16 exposures per ft of boring, sufficient overlap from picture to picture is obtained.

Suspending the camera from the ground control mechanism is a three-conductor cable, armored externally by reverse-lay preformed steel wire which prevents cable twisting. The tensile strength of this cable is 2700 lb.

On the ground surface the camera is controlled by a lowering device, Fig. 3, which consists of a pay-out reel *(c)* with a capacity for 500 ft of camera cable, level-wind control *(b)* for the cable guide reel *(h)* geared to a 16-notch pulsing wheel and depth counter

(i and j), and a hand-crank and gear drive *(g)* to the cable pay-out reel for lowering and raising the camera. Attached to the lowering mechanism is a small power supply cabinet *(a)* which transforms 117 v ac to three dc circuits of 450, 50, and 50 v, respectively. The highest voltage is transformed to 15,000 v in the flash tube circuit of the camera; one 50-v circuit is connected to the camera motor drive and synchronizing relay; the other 50-v circuit provides the synchronizing pulse. When a film is completely exposed, the individual pictures are projected and studied as a series of separate still photoflash images and not as a moving picture film.

Pictures are taken during the ascent or retrieving of the camera, in as much as cable tension is more constant and the motion of the camera steadier. A dummy camera of the same weight and dimensions is lowered and retrieved before the real camera is risked in the boring. Since the camera pulsing wheel is geared into the crank and gear-drive of the cable reel and guide reel, the time interval between picture exposures is controlled by the speed at which the hand crank is turned. Best results are obtained when the exposure interval is 2/3 sec. With a capacity for 25 ft of 16-mm film, 75 ft of boring may be photographed continuously before reloading is needed.

The camera is designed to withstand a hydrostatic head of 500 ft. Pictures obtained in dry and water-filled borings are equally good.

All the camera equipment is portable into any site where diamond core-drilling equipment can enter. With the aid of temporary cribbing and leveling screws *(d)* attached to the frame, the ground-control mechanism can be leveled and made ready for operation a few minutes after the equipment is uncrated at the site. A portable 117-v ac gasoline generator supplies the necessary electric power when other sources are unavailable.

No less unique than the camera, from the standpoint of optics, is the device that was developed to project the continuous and overlapping pictures obtained by the camera. This unusual projector transforms the pea-sized doughnut-shaped images, which the camera's cone mirror impresses on the film, into true-to-scale cylindrical images. This is three-dimension picture projection in its truest sense. The cylindrically projected image simulates a 1-in. section of the inside of the boring. Each picture presents accurately the depth, orientation, and dimensions of every feature of the rock and, on color film, discloses much information as to its composition, texture, and soundness. The advantages of the pictures over the rock cores are that 1—conjecture re-

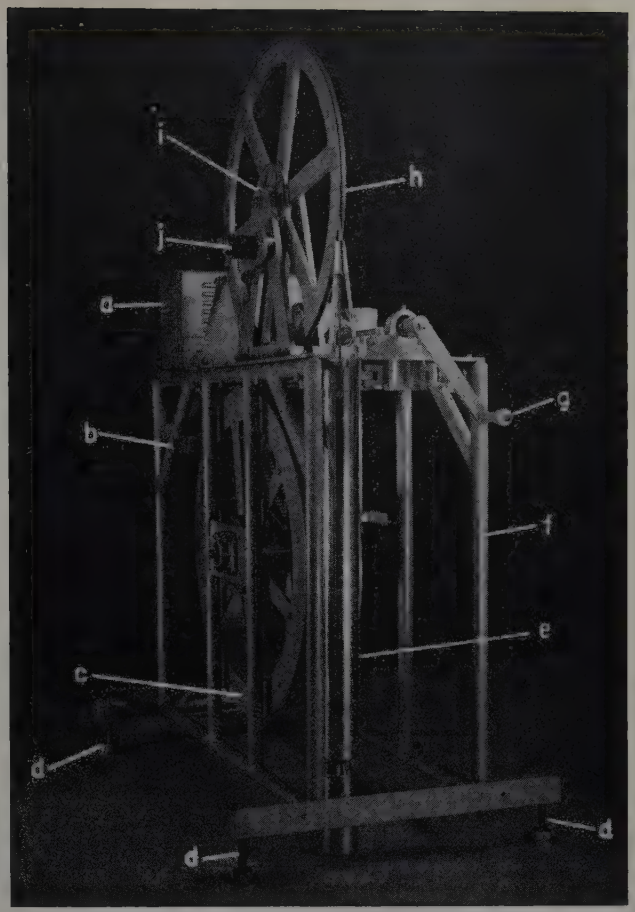
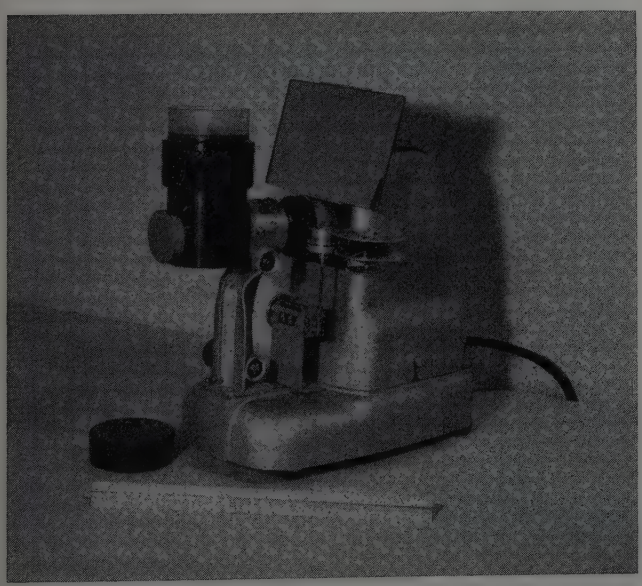


Fig. 3—The borehole camera machine, showing control frame *f* and camera proper *e* ready for photographic operation. Note cone mirror near lower end of camera tube. Camera tube diam is 2¾ in.



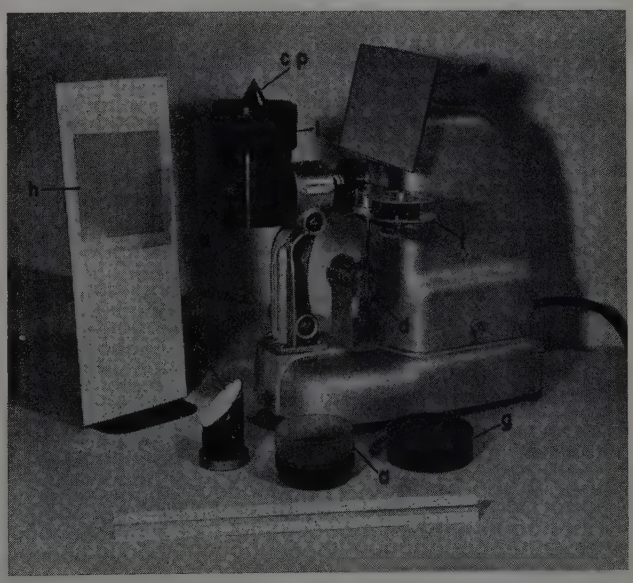


Fig. 4 (right)—This borehole camera projector is used to view transparencies taken by the borehole camera, which photographs a 360° arc of the walls of a 3-in. drillhole. Fig. 5 (left)-The same projector, with 45° plane mirror, *c*, and cylindrical ground-glass screen, *a*, removed for plane image projection on screen, *h*. Reference numbers are covered in text.

garding core losses is eliminated, and 2—the orientation (dip and strike) of faults, fractures, contacts, bedding planes, and other structural features of the rock can be determined easily and accurately.

The cylindrical projector, as shown in Figs. 4 and 5, is a converted Viewlex 35-mm projector, adapted to support either the conventional 5-in. lens for large plane-image projection or the L-shaped projection tube developed for combined cylindrical-image and small plane-image viewing. A 500-w lamp supplies the required light. In cylindrical projection, light passes through the small circular image on the film and is projected by the lens to the 45° plane mirror (*c*) at the base of the vertical arm of the L tube (*l*). The inclined mirror in turn reflects the circular image upward to a clear-glass conical prism *(cp)*, slightly larger than the cone mirror of the camera. By refraction the prism transforms the plane image into an inverted cone of light intercepted by a surrounding frosted-glass cylindrical viewing screen *(a)* of the same diameter as the original boring. The picture is inverted on the film spools before projection so that it may be viewed in proper orientation on the screen.

Compass orientation of the cylindrical image is obtained by lining up the north arrow visible at the top of the conical prism, Fig. 5, with 0 degrees on the adjustable azimuth ring at the base of the viewing screen. The trend and inclination of fractures or bedding planes in the rock can be measured quickly by a straight-edge and protractor. A counter *d* geared into the right-hand film spool *f* records the correct depth of each picture. Thus while the camera transforms a section of the interior of the borehole into a plane image on the exposed film, the new projector reverses this process by transforming the plane image into its original cylindrical form.

A most convenient feature on the new projector is its adaptability to plane-image projection. Retracting the inclined mirror from the aperture (*ca*) projects a true-scale inverted plane image of the boring to a flat screen, see Fig. 5. This projection is merely an enlargement of the small picture on the film. It is used exclusively for editing the film strip, since every feature of consequence to the geologist can be detected rapidly before special sections of film are subjected to detailed study on the cylindrical screen.

To assist the reader in visualizing the relationship between the plane and cylindrical images, attention is called to the diagrams, Figs. 6a-6c. Fig. 6a is a core specimen of granite gneiss obtained from a drillhole along the Rappahannock River, Fig. 6b is the borehole camera film image of the section of the boring corresponding to the bracketed section of the core, and Fig. 6c is the cylindrical projection of the plane image. In all three figures, *J* is a prominent rock fracture paralleling the lamination of the rock, while *f* is a secondary branching fracture. While the composition and soundness of the rock could be ascertained readily on Fig. 6b, the trend, inclination, and dimensions of the fractures could be viewed in their undistorted relationships only by the cylindrical projection, Fig. 6c.

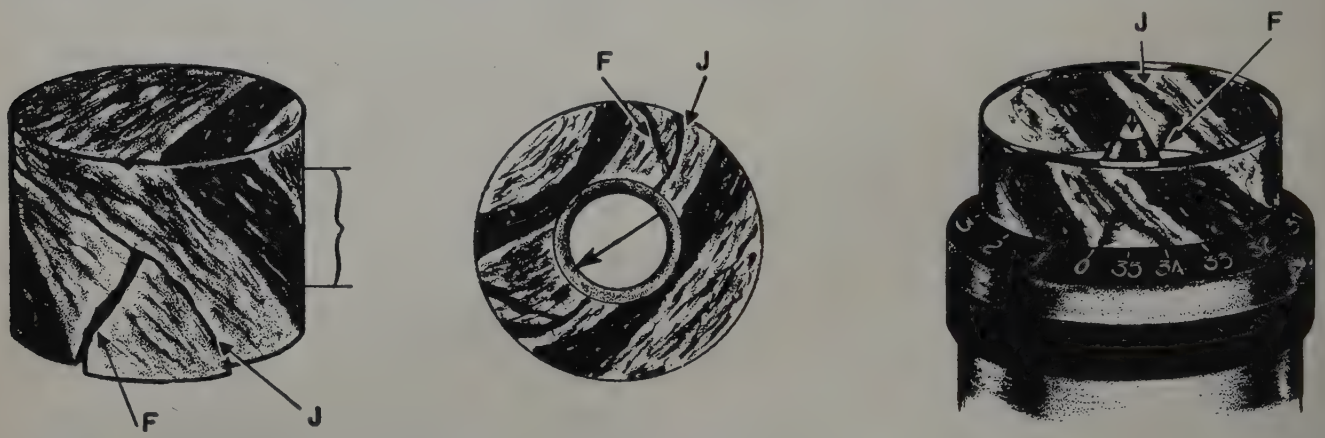


Fig. 6a (right)—Segment of 2⅛-in. core of granite gneiss. Fig. 6b (center)—Borehole camera film image of section of 3-in. boring from which core was extracted. Fig. 6c—Cylindrical projection of film image. Bracket on 6a shows depth range of 6b and 6c.

Assuming that both pieces of rock core below the fracture *J* were lost during drilling operations, the geologist examining the core might conclude logically that either a sizable void occurred at this elevation or that the rock was too highly disintegrated to permit complete sample recovery. The new borehole camera would solve the problem immediately by disclosing the type and structure of the rock at this point and the precise direction and slope of the two sets of fractures responsible for loss of the core sample.

The Chief of Army Engineers is continuing research designed to improve the efficiency and versatility of the camera and the projection device.

Corrections

In the April 1954 issue: TP 3768B. Flotation of Oxidized Zinc Ores. By M. Rey, G. Sitia, P. Raffinot, and V. Formanek. On p. 420, col. 2, under Conclusions, the first sentence should read as follows: After six years of study in the laboratory and three years of mill operation treating more than 10,000 tons of ore, it is held that flotation of oxidized zinc ores by the combination of sodium sulphide and primary amines is an efficient process and well under control.

In the May 1954 issue: TP 3743B. Flotation and the Gibbs Adsorption Equation. By P. L. de Bruyn, J. Th. Overbeek, and R. Schuhmann, Jr. On p. 520, col. 1, Eq. 4 should read as follows: $\Sigma_i n_i d\mu_i = 0$

Change to Rotary Blasthole Drilling in Limestone Increases Footage, Cuts Time, Saves Manpower

by D. T. Van Zandt

IN the late 1920's rotary drills began to replace the churn drills in the petroleum industry, but until the middle 1940's the churn drill was the only widely accepted means of drilling large-diameter blastholes for quarry operations.

The Calcite plant of the Michigan Limestone Div., U. S. Steel Corp., was one of the first to experiment with rotary drills for quarry blasthole drilling, and the first to employ compressed air on a fully rotary rig to cool the bit and raise the cuttings to the collar of the blasthole.

The Calcite plant operates a limestone quarry near Rogers City, Mich., in the northern part of the lower Michigan peninsula. The formation quarried, a portion of the middle Devonian series, is the Dundee limestone, which is uniform, seldom massive, and characterized by definite bedding planes. The dip is southeast, 40 ft to the mile. Quarry faces vary from 20 to 116 ft in height. Vertical blastholes are used entirely, from three to five rows of holes being drilled parallel to the working face, spaced 18 ft apart with 18-ft burden and drilled 6 to 8 ft below shovel grade. Quarry operations coincide with the navigation season on the Great Lakes, as the bulk of the stone is transported by lake carrier. The normal operating season runs from April to December, the remaining time being devoted to stripping operations and plant and equipment maintenance.

In the following discussion drilling rates mentioned refer to overall drilling time and include all operations such as moving from hole to hole, penetration and extraction of tools, and routine maintenance. Time consumed by such factors as power delays and major machine repair is not included in drilling time unless otherwise stated. Figures cover only operations at this one plant in the formation mentioned. Needless to say, a very different set of figures could be obtained in a different formation. However, the comparison of footage obtained with churn drills and rotary rigs in this particular formation has been used as an indication of what might be the expected performance of rotary rigs in other formations.

Prior to 1950 the bulk of the blasthole drilling at the Calcite plant was done by electrically powered churn drills. Both crawler and wheel-mounted rigs were used. These machines, which mounted a 22-ft drill stem of 4½ in. diam and a spudding type of bit 2 to 4 ft long, drilled a hole of 5⅝-in. diam. Average drilling rate of these rigs in the Rogers City formation was 8½ ft per hr.

In 1946 one of the first rotary blasthole drills offered to the quarry industry was put into use on an experimental basis. This machine, known as the Sullivan Model 56 blasthole drill, Fig. 1, was on 16-in. crawler pads and electrically powered at 440 v. The drill bit, a Hughes Tri-Cone roller bit of 5⅝-in. diam, Type OSC, was threaded into the end of the 4-in. square hollow drill rod or stem. These drill rods were 20 ft long with female threads on one end and male on the other to allow for addition of the desired number of rods for drilling holes of various depth. Rods were handled by a single drum hoist geared to the main drive motor and racked by a 30-ft derrick or mast when not in use. The cable from the hoist drum fed through a crown block on the top of the derrick back to the water swivel mounted in the top end of the drill stem in use. This cable remained attached during drilling operations and was used to hoist the tool string from the hole. Down pressure was applied to the tool string by means of a pair of 4-in. diam hydraulic cylinders acting on the drill chuck holding the drill rod. The first chuck consisted of flat jaws which gripped the flat sides of the stem. These jaws were controlled by set screws forcing them into contact with the drill stem. As these set screws had to be loosened and tightened by hand with each stroke of the hydraulic feed cylinders, there was great delay. For this reason the semi-automatic chuck was developed which automatically gripped the stem on the downward stroke but released for retraction of the hydraulic feed cylinders. Rotation was imparted to the tool string by a rotary table acting on the chuck and geared to the main drive motor through a separate gear train and clutch. A positive displacement water pump, mounted on the drill, fed water through a system of pipes and hose into the water swivel mounted on the top of the drill rod and through the rod and bit, washing the drill cuttings to the collar of the hole. Where water was scarce, provision was made to settle out the cuttings coming from the collar of the hole and re-use the water. Where water was abundant the stream coming from the hole was wasted. Drilling rate with this machine was about 20 ft per hr and bit life 1600 ft of hole.

While this rate was more than twice that obtained with the churn drills employed, the problem of water supply and drill cuttings disposal rendered the machine impractical from an operating standpoint. Consequently it was used only in that part of the operation for which water was easily supplied, when the character of the formation made it least difficult to wash cuttings away from the collar of the hole.

In October 1949 it was suggested that drill cuttings be removed by compressed air, long used for this purpose on pneumatic drills, and collected at the collar by suction. Thereafter, the water pump on the Sullivan 56 was replaced by a 500-cfm air compressor and a trial run made. Air pressure at

D. T. VAN ZANDT is with the Michigan Limestone Div., U.S. Steel Corp., Rogers City, Mich.

Discussion on this paper, TP 3817A, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, Feb. 16, 1954. New York Meeting, February 1954.

the water swivel was held between 30 and 50 psi and rotation varied from 35 to 105 rpm. Pressure of 500 psi was maintained on the hydraulic feed cylinders. The same bit as that used with water was employed, a Hughes Tri-Cone of 5⅝ in. diam. Several holes 40 ft deep were drilled.

Because the equipment used was cumbersome to move, during these first tests the points checked were primarily penetration rate and bit life. Penetration with a new bit proved to be 15 in. per min or 75 ft per hr. With water the penetration rate had never been more than 38 ft per hr. After penetration rate had been carefully checked over 24 hr of drilling time the tests were temporarily stopped. At this point the bit showed little sign of wear; the bearings were in very good shape and the teeth only slightly worn.

Because of the favorable results obtained thus far, it was decided to remodel the Sullivan 56 for permanent use. Calculations by engineers of Hughes Tool Co. showed that an air velocity of some 3000 ft per min should be maintained in the drillhole to raise the cuttings successfully and constantly offer a clean surface to the bit. As the bearings in a 6¼-in. Tri-Cone bit were more suited to the operation, this size was recommended and the orifices modified to give better cooling to the bearings and to keep the bottom of the hole clean.

Following the recommendations of the Hughes Tool Co. and the Joy Mfg. Co., a 365-cfm Joy air compressor and a rotoclone, Model 8 D, manufactured by the American Air Filter Co., were mounted on the Sullivan 56 drill and the tests continued. By use of orifices in the bit, the only means of throttling the air flow, an operating pressure of 68 psi was maintained at the swivel. Down pressure on the tool string was varied from 400 to 650 psi, or a maximum of 11,000 lb weight on the bit, and rotation was varied from 35 to 105 rpm. Best operating conditions

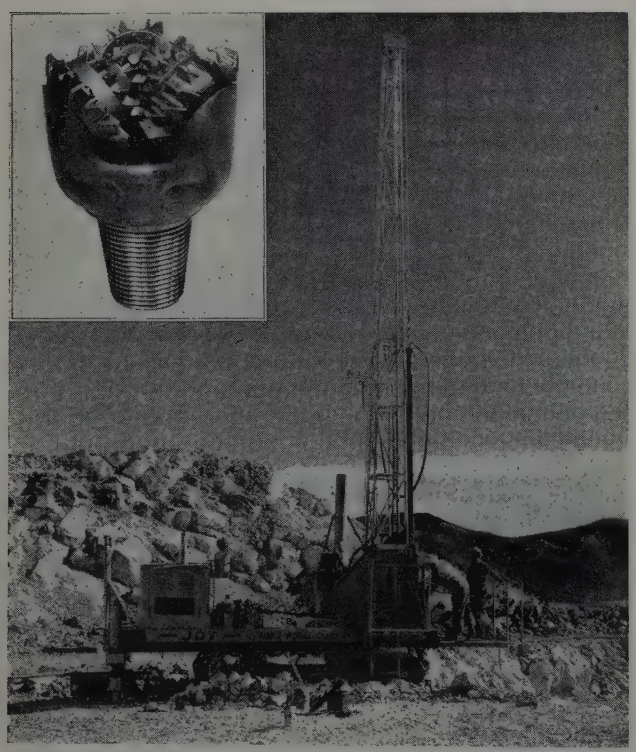


Fig. 1—First rotary rig tried was of same type as the Sullivan 56 shown here. Drill was remodeled to incorporate 365-cfm compressor and rotoclone. Inset: Type OSC Tri-Cone rock bit furnished by Hughes Tool Co. for rotary blasthole drilling.

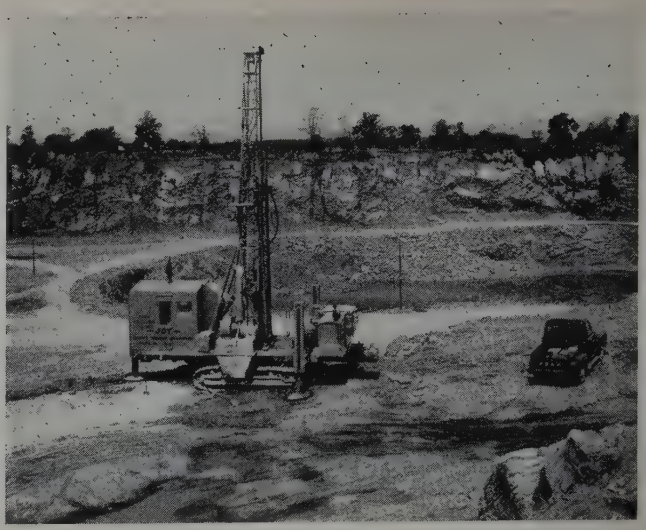


Fig. 2—The Sullivan 58, above, weighs 25 tons, handles 30-ft instead of 20-ft rods, and has 48-in. feed.

were found to be at 80 rpm and 500 psi down pressure. With a new bit the drill was operated for 137 hr under these conditions, moving time included. Total footage drilled during this period was 5227 ft, an average drilling rate of 38 ft per hr, collar to collar. Penetration rate with 100 ft of hole on the bit was 75 ft per hr. At 5227 ft of hole on the bit, penetration rate had dropped to 42 ft per hr. At this point the bit was carefully inspected. The bearings were in fair condition, all cones turning freely but feeling rough. The teeth showed considerable wear but not serious loss of gage.

The bit was replaced with a new one of the same type and testing continued under the same conditions. Bit life of 4905 ft was obtained after a period of 130 hr of operation. Bit condition was essentially the same as that of the bit previously run.

During the latter part of the period the Sullivan 56 was being tested and modified at the Calcite plant, the Joy Mfg. Co. developed a heavier drill built around the same general principles as the 56, but with several major modifications. This drill, known as the Joy 58, weighed about 25 tons as compared with the 12 tons of the 56. This newer type, Fig. 2, used either diesel or electric power. The chuck on the new rig was located in the center of the machine rather than at the end so that the weight available to the tool string, applied through a pair of 6-in. diam hydraulic cylinders, could be about double that applied to the 56. A 40-ft mast or derrick allowed the use of 30-ft drill rods instead of the 20-ft sections standard on the 56. The new rig was also crawler-mounted, and in addition carried three hydraulic jacks for use in leveling the drill. The derrick was also raised and lowered hydraulically. A modified semi-automatic chuck operating over a 48-in. feed range replaced the 30-in. feed range of the 56. Controls were in the operator's cab located back of the chuck. This cab was so arranged as to give the operator clear vision of all operations. A 365-cfm air compressor was mounted on the forward end of the drill, replacing the water pump originally carried. A Model 8 D rotoclone on the side of the machine collected the cuttings from the collar of the hole, drawing them through an 8-in. flexible hose and discharging them alongside the drill. The machines operated at the Calcite plant carried a bank of 4000/440-v transformers behind the operator's cab. The transformers were Y-con-

nected with grounded neutral and instantaneous breakers, as is general practice in the quarry. Lighting was on 110 v through a separate dry-type transformer. Each machine was equipped with 1500 ft of No. 6, three-conductor, rubber-covered trailing cable which tied into the 4000-v quarry system.

Four of these machines were purchased by the Michigan Limestone Div. for use at the Calcite plant in March 1950. The drills were put into use immediately following their delivery. While these machines were in general similar to the Sullivan 56, the added weight available to the tool string led to considerable experimentation to find the best operating conditions. Extensive tests conducted by the Hughes Tool Co. proved the penetration rates shown in Fig. 3 to be possible. Bit life was found to be a function of bit bottom time and pressure.

Taking into consideration penetration rate, bit life, drill maintenance, and overall drilling rate, a pressure of 550 psi at 86 rpm was found to give best overall results. Under these conditions the collar-to-collar drilling rate, based on records covering four 9-month operating seasons on the four machines, was found to be 45 ft per hr with an average bit life of 3800 ft.

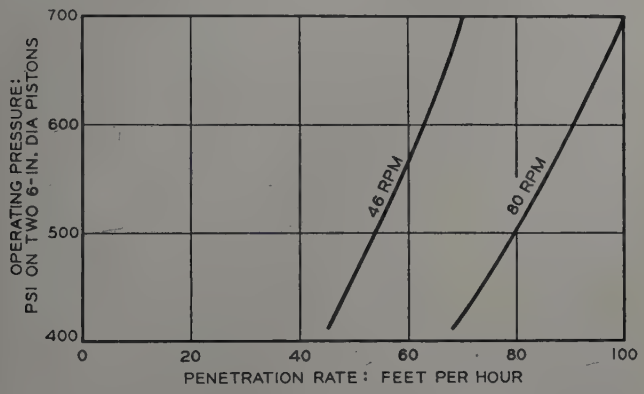


Fig. 3—Variation of penetration rate with down-pressure at various operating speeds.

Previous to the purchase of the rotary drills, the drilling at the Calcite plant was done by churn drills. In the years of 1948 and 1949 the drilling requirements of this plant were slightly over 500,000 ft of blasthole per operating season. To obtain this footage 16 churn drills were operated 16 hr per day during the operating season and five drills operated 8 hr per day during the stripping season. During the years 1950 through 1953 an average of 615,000 ft of blasthole per season has been required. Employment of four Joy 58's and one Sullivan 56 drills 16 hr per day during the operating season has supplied this footage. The operators of the new machines had used churn drills for an average 23 years and averaged 51 years of age. Their breaking-in period on the rotary drills was supervised over a one-month period by a Joy service engineer. During the four years the drills have been in operation the drilling department has not had one lost time accident on the rotary machines.

Because stripping operations completely remove the overburden and loose stone from the limestone being drilled, there has been very little experience of drilling loose or unconsolidated material at the Calcite plant. During the time the churn drills were in use steel casing was used in all holes, sometimes to a depth of 20 ft. No casing is used in the rotary drillholes, even in the softest formations. Where the collar is loose, cardboard casing 2 ft long is inserted after the drill has moved off the hole. This is done mainly to assist the blasters in loading the holes, as the casing furnishes a smooth collar through which to load. No attempt is made to reclaim the casing.

Some water is encountered as drilling grade in some areas lies 6 to 10 ft below the local water table. In drilling these areas the hole generally remains dry as long as the air stream through the tools is maintained. If there is occasion to cut off the air when the bit is below water level the hole fills with water in 2 to 5 min. During this time it is possible for cuttings to be forced up into the body of the bit. To prevent this, a screen is located in the bit to keep the larger particles from entering the air jets serving the bearings. When drilling is resumed the water and mud are blown to the collar of the hole. Under these circumstances it is found inadvisable to use the dust collector as the moist cuttings block the passages to the suction fan.

During freezing weather and also when the humidity is very high there has been difficulty in keeping the passages through the dust collector clear. Several access doors were added to the standard roto-clone to facilitate cleaning when necessary. When drilling is done during freezing temperatures anti-freeze solution introduced to the air stream beyond the compressor receiver prevents freezing in the dust collector.

Calculations have shown that an air velocity of some 3000 fpm must be maintained within the blasthole to raise the cuttings to the surface successfully. Experience at the Calcite plant has shown this to be true with a 6¼-in. bit mounted on rods 4 in. square with a penetration rate up to 75 ft per hr. Where penetration rate is higher than this, it is possible to make cuttings faster than they can be removed and a stuck tool string is the result. When this occurs, down pressure must be reduced to zero and rotation and blowing continued. If this does not free the tool string, the tools may have to be raised during rotation, possibly by a combination of hydraulic pressure and hoist cable.

It is to be noted that the overall drilling rate with the Joy rotary drill is about half the actual penetration rate. This is due to several factors, i.e., moving from hole to hole, leveling the machine, retracting the chuck over the available feed, adding drill rods, removing drill rods, and lowering the machine upon completion of the hole preparatory to moving to the next hole. In addition to these operations there is the routine maintenance such as lubrication. These factors and the time they take have been included in the drilling rate figures quoted and amount to about half the available time in a shift. It has been proved that in any given formation under set conditions of rotation and weight on the tool string, penetration rate will remain constant. To increase the overall drilling rate the time, motion, and labor involved in getting the bit on bottom and actually drilling the hole must be decreased. It is encouraging to the quarry operator to see that several companies are attacking this problem. At the Calcite plant it is seen that with rotary drills more feet of blasthole are being drilled in less time with fewer men than were being drilled with churn drills. The advent of the rotary drills is the first major change in large-diameter blasthole drilling in some 30 years. Progress is by no means at an end. With the developments now in sight it is quite possible that in the space of a very few years the present rotary rigs will have become as obsolete as the churn drills.

Relationship of Geology to Underground Mining Methods

by George B. Clark

Many basic engineering principles of all four phases of mining operations, namely, prospecting, exploration, development, and exploitation, can be analyzed better in terms of quantitative geology. Geological data from both field and laboratory will also complement scientific methods now being developed.

THE geological data emphasized so successfully in prospecting for new deposits, that is, structural controls, strength of solutions, and type of mineralization, are basically those required for successful exploitation. In the mining of newly discovered deposits the most economical methods should be employed as early as possible to keep the overall cost per unit produced at a minimum and to permit maximum extraction of valuable minerals. A crucial question is: How can geological data be translated into useful quantitative results which will aid in achieving this end?

H. E. McKinistry[1] has suggested that a solution may be reached in one of two ways: 1—the usual approach, use of judgment based on experience; or 2—mathematical calculations and tests on models, both subject to certain limitations. He also suggests that in addition to better use of geology more case data and theoretical data are needed on which to base sound judgment. Further research, therefore, is necessary. Perhaps in this field the emphasis should be on more specialization in mining methods and ground movement by men with thorough training in physics, engineering, geology, and underground mining. These specialists would be equipped to point out the most economical and scientific methods of exploitation.

Selection of a stoping method is governed by the amount and type of support a deposit will require in the process of being mined, or by the possibility of employing the structure of the deposit to advantage in mining the ore by a caving method.

In addition to these factors there are others which almost invariably influence the choice of an economical method of mining:[2] 1—strength of ore and wall rocks; 2—shape, horizontal area, volume, and regularity of the boundaries of the orebody, and thickness, dip and/or pitch of the deposit and individual ore shoots; 3—grade, distribution of minerals, and continuity of the ore within the boundaries of the deposit; 4—depth below surface and nature of the capping or overburden; and 5—position of the deposit relative to surface improvements, drainage, and other mine openings.

In the final analysis it is usually necessary to disregard the less important of these factors to satisfy the requirements of the more important. Because of the variation of geological conditions throughout and surrounding the deposit, no mining method will be everywhere ideally applicable to the conditions encountered in one ore deposit.

The immediate problem is to interpret the above physical characteristics of deposits in terms of geological characteristics. Very few quantitative geological data are available on the factors related to a choice of mining methods. However, there are many descriptive data in mining and geological literature which collectively show how important an effect details of geology have upon all phases of mining operations.

The following categories of basic mining methods were investigated to establish the geological factors that have affected their successful application: 1—open stopes with pillars; 2—sublevel stoping; 3—shrinkage stoping; 4—cut-and-fill stoping; 5—square-set mining; 6—top slicing and sublevel caving; and 7—block caving. It should be noted that the first five of these methods are listed in the order of increasing support requirements. Mines were selected as examples only where geological descriptions were complete enough to warrant their use.

A study of the geological factors involved in mining operations led to a choice of the following classifications, employed in Table I: 1—structural type of orebody; 2—dimensions (geometry); 3—country rock (type); 4—faulting, folding, and fracturing; 5—alteration of ore and rock; 6—type of mineralization; and 7—geological factors determining mining method (summary). Of these factors only one yielded results that can be defined from available data in a quantitative manner, i.e., dimensions of the deposit.

These are the most reliable guides that can be used in selection of suitable mining methods. They are, in general, the properties of geologic structure most difficult to evaluate by studies of models, photoelastic studies, and other laboratory methods, all of which are at present more limited in their applications than the geologic method. Application of geology has proved a reliable guide in other phases

G. B. CLARK, Member AIME, is Professor of Mining Engineering, University of Illinois, Urbana, Ill.

Discussion on this paper, TP 3834A, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, Oct. 30, 1952. New York Meeting, February 1950.

of mining and requires only an extension of basic principles for application to stoping operations.

Geologic data for 52 mines were compiled, tabulated, and analyzed. Table I illustrates the method of tabulation. The mines listed are not, of course, necessarily representative of all those employing a given mining method.

Three examples, one of which is given under the classification of open stopes in Table I, were taken as representative of bedded deposits amenable to open-stope mining with pillars. They are noted for simplicity and strength of structure, competency of the beds, absence of extensive faulting, and absence of destructive alteration. In spite of the fact that dimensions of the deposit are variable, the open-stope method has uniform application throughout the deposits as long as the ore and rock are strong enough to support large openings. The placing of pillars is governed almost entirely by the presence of minor faults, fractures, or weak beds. Where these planes of weakness do not occur the pillars may be spaced regularly. It is noteworthy that many deposits may occur in sedimentary rocks which have been metamorphosed. In the case of the deposits in the Tri-State district, the rock was strengthened considerably by the presence of silica which resulted in the conversion of the limestone to chert and flint. In most cases the bedding planes, although inherently weak in sedimentary rocks, did not cause problems of support unless they were accompanied by faults and fractures.

Mine openings in shale almost invariably require timber. If water is also present the shale becomes muddy, having the consistency of soft clay. If it is dried out by dry currents of air in the mine atmosphere, it tends to slough and spall.

Water courses are also consistent sources of trouble. Even if they are dry they represent planes of weakness. If they are active, they create pumping problems.

Sublevel Stoping

Employment of the sublevel method of stoping requires, among other characteristics, a strong ore which will serve as a strong roof and offer safe footing for men and equipment on benches that have been undercut by mining. Massive sulphide ore which has not been subjected to fracturing or alteration processes such as oxidation is almost ideal for this type of mining. The sulphide minerals seem to have a capacity for filling small fractures as well as large ones, providing a mineral complex, even in complete replacement processes, which is very competent and strong. The healed fractures vary from those of microscopic size to faults and even cavities of fairly large dimensions.

Strong walls are found in rocks of all three genetic types. Fine-grained rocks are generally very competent, even when slightly fractured. In all the examples studied there is a remarkable lack of extensive post-mineral faulting, fracturing, and alteration processes that weaken the rock structure.

The large size of the ore deposits is a desirable feature, even in the case of the massive and irregular lower H orebody in the Horne mine, where the sulphide ore itself often constitutes the walls of the stope. In the case of this deposit, the silicification of the rhyolite breccias has played an important part in strengthening the enclosing rock structure.

Shrinkage stoping is classified by some as an open-stope method of mining and by others as a supported stope method. Each classification is probably justified because the back of the stope, which is usually ore, is unsupported during the process of mining, while the walls are supported by broken ore whether they need immediate support or not. Deposits mined by this method are on the borderline between those that require support during mining and those that do not. In terms of strength of ore and walls, this must mean that the ore should be strong in any case, while the walls may be weaker than the walls of deposits mined by sublevel stoping.

A study of 14 mines shows that there has been little if any significant destructive alteration of the ore or rock, but there is more evidence of dynamic movement of the rock in the deposits mined by shrinkage stoping, especially some post-mineral faulting.

In some cases, as at the Hollinger and Wright Hargreaves mines, the size of the veins was the determining factor, only narrow veins being mined by this method. At the United Verde mine a massive sulphide body, non-ore, serves as the host rock for the ore deposits. Unless disturbed by faulting and fracturing it is very competent, as are similar bodies (ore) at Tennessee Copper Co. (sublevel stoping).

Cut-and-Fill Stoping

Cut-and-fill stoping is employed successfully in those deposits that display a degree of structural weakness just one step further removed from those that are strong and accessible by shrinkage methods. It is common to find both these methods of mining used in the same mine to extract the ore from different sections of the same deposit. Many mines use three methods of mining in the same deposit, that is, square-set stoping in addition to the two noted above.

Cut-and-fill stoping requires relatively strong ore that will need no support during stoping operations. It is used to advantage where the ore is strong but the walls so weak that shrinkage methods cannot be used without causing dilution of ore.

The geological reasons for use of cut-and-fill are summarized as follows:

1—Weakening of rock structure by pre-mineral faulting.

2—Weakening of rock structure by post-mineral faulting.

3—Further weakening of rock by hydrothermal and hydrometamorphic processes.

4—The absence of silicification in certain areas of a deposit.

5—The absence of other types of healing mineralization.

6—Irregularity of outline of deposit, which requires a selective method of mining.

7—Irregularity of grade or lack of uniformity of grade of ore, which also requires a selective method.

8—Rapid oxidation of ore, preventing use of shrinkage stoping which *stores* ore in stopes for long periods of time. This is true for ore that is to be treated by flotation where oxidation of sulphides interferes with the flotation process.

The factors affecting the use of square-set mining are as follows:

1—Grade of ore must be high enough in grade to pay for the large amount of timber required.

2—Physical character of ore and rocks. In nearly all the mines using this method the enclosing rocks are broken and altered. Structurally weak ore and rocks usually go together, although this is not always the case.

3—Size, shape, and dip of deposit. The method is

Table I. Geological Factors Affecting Mining Operations

Method and Mine	Structural Type of Ore Body	Dimensions	Country Rock	Faulting, Folding, Fracturing, Etc.
Open Stopes with Pillars Southeast Missouri	Nearly horizontal bedded deposit	Thickness 7 to 200 ft, width 800 ft, and length 1200 ft	Dolomitic limestone (Overburden 300 to 500 ft, limestone and shale)	Few important faults
Sublevel Stoping Horne mine, Noranda, Que.	Large, irregular nearly vertical body of massive sulphides, occurring as a replacement of brecciated rhyolite	*H* orebody: length 600 ft, width 40 to 450 ft	Rhyolite, brecciated	Faults of small displacement and shear zone-faults are of varying intensity
Shrinkage Stoping Hollinger mine, Porcupine, Canada	(See last column) Veins along fractures	Widths to 75 ft	Altered and distorted igneous and sedimentary rocks: Greenstone, porphyry and *pillow lava* schistose parallel to strike of veins	Post-mineral faults of small displacement. Considerable dynamic action has caused distortion, shearing, and schisting
Cut-and-Fill Stoping Creighton mine, Ont., Canada	Lenticular deposits in shear zones in norite and granite	Up to 1000 ft long, 300 ft wide. Dips: 35° to 60°	Norite hw, and granite fw	Brecciation caused by pre-mineral faulting
Square-Set Stoping Tintic Standard, Tintic district, Utah	Replacement ore—sulphides and oxides in massive limestone beds	Massive, 20 to 200 ft thick	Limestone, shales, and quartzite	Intense pre-mineral and post-mineral faulting
Top Slicing Blueberry mine, Michigan Marquette Range, Michigan	Tabular type deposit, nearly vertical	Average width 50 ft, length 200 ft. Dip: 75° to vertical	Slate fw, cherts, and jaspers	Pre-mineral folding and faulting
Sublevel Caving Gogebic Range, Eureka Asteroid mine, Michigan and Wisconsin	Concentration of hematite in pitching troughs formed by intersection of dikes with fw quartzite or impervious slates	Irregular in shape from few to several hundred feet in width and thickness; from several hundred to several thousand feet in length	Hw-bands of slate and partly leached cherty iron formation, breaks into slabs which tend to arch	Numerous faults of all strikes and dips
Block Caving Climax mine, Climax Molybdenum Co. Climax, Colo.	Top portion massive and dome-shaped; bottom portion elliptical ring	Elliptical section 3000 ft and 2800 ft on two axes. Mineralized zone or ring 400 ft wide. Capping: 0 to 300 ft	Silicified schist, granite, and early porphyry Rock classed as: 1—Very strong 2—Moderately strong 3—Moderately weak 4—Very weak	Hard granite cut by fractures Orebody intensely fractured by post-mineral and pre-mineral faulting in places

very flexible and can be used in almost any size of deposit regardless of its shape and dip.

4—Effect of ground movement. There may be some settling in the fill, and there is squeezing and subsidence of country rock.

5—Availability and cost of timber, labor, and rate of oxidation.

6—Safety of mining operations.

The square-set method is the one usually employed when other methods, exclusive of caving, fail to satisfy conditions for safe mining.

For both top slicing and sublevel caving methods of mining, the most necessary requirement of the structure is a weak capping that will cave when it is undermined. This feature is found in almost all examples studied and is due either to the presence of weak members in the overlying formation or to the presence of planes of weakness. In any event the caving formations should not *key* and arch to such an extent that sudden collapses will create dangerous mining conditions. Top slicing is more adaptable to deposits of large horizontal extent,

Table I Continued

Alteration of Ore and Rock	Type of Mineralization	Geological Factors Determining Mining Methods
Little local alteration	Disseminated galena in limestone and shale	1—Flat bedded deposit —simple geometry 2—Simple undisturbed structure. Few faults and fractures 3—Strong ore and competent rock
Silicification of breccias Sericitization: limited to areas where it does not critically affect mining Chloritization	Sulphides carrying Cu and Au ore is unusually strong and compact in character and stands well Occasional slips in ore; little gangue material	1—Silicification of breccias strengthened rocks 2—Massive sulphide ore has but few slips and is very strong 3—Ore does not oxidize easily before flotation 4—Little effective weakening alteration of country rock or orebodies
Dynamic alteration of rocks	Quartzite, pyrite, Au fracture patterns determine strength of the ore	Only minor post-mineral faulting and hydrothermal alteration Shrinkage stoping used only in veins thinner than 8 ft and with strong walls
Pyritization-sulphides have cemented some breccias	Massive and disseminated sulphides in morite and granite	Cut-and-fill used where: 1—Weakness due to shear 2—Irregular orebodies 3—Rock bursts in granite 4—Pressure due to depth of mining operations 5—High-grade ore requiring selective method
Intense hydrothermal alteration and oxidation of both ore and limestone in and near deposit	Lead-silver ore, sulphides, and oxides	Intense fracturing, alteration and oxidation weakened structure so that immediate support is required. Previous methods also weakened overall structure.
Some country rock metamorphosed to chert and jasper	Hematite ores, secondary concentration in brecciated zone of iron bearing sediments	Top slicing used in wide ore, otherwise mined by sublevel stoping Capping caves readily; ore is strong enough to stand temporarily
(?)	Concentration of hematite in pitching troughs	1—Triangular shape of orebodies causes capping to cave slowly 2—Soft weak ore 3—Fairly strong capping which slabs when unsupported but delays caving temporarily
Sericite slips in granite Silicification intense at fw to slight at hw Capping shows surface alteration and less silicification	Molybdenite in fractures and dissemination in quartz veinlets	Massive orebody and capping are fractured and altered and weak enough to cave. Weakness due to both post-mineral and pre-mineral faulting. Ore occurs in very weak to moderately strong rock, all of which may be caused to cave.

while sublevel caving can be employed to mine deposits more irregular in outline. In both cases the ore should be moderately weak, but strong enough to stand temporarily. This characteristic may be due to the inherent character of the mineral complex itself or to alteration and fractures.

Iron deposits in the Great Lakes region are amenable to extensive application of one of the above methods, both because of the character of capping and the geometry of the deposits.

The work of King at the Climax Molybdenum[3] mine is one of the few published attempts to evaluate the strength of mine rock in a quasi-quantitative manner. The strength of the rock and the ore are defined in terms of the spacing of fractures, type of mineralization in the fractures, and silicification and alteration of the constituent minerals. The original composition of the rock and the type of alteration are also important. Silicification and kaolinization together almost universally indicate weak rock structure. Strength and composition of mineralizing solutions, pre-mineral and post-mineral faulting, and composition of the local rock all contribute to strengthening or weakening the rock structure.

At Climax the rock formations are strong as compared with those of other block caving mines. However, the relative positions of the four classes of rock, together with the fact that the ore is not very strong, make the deposit adaptable to block caving methods.

In the *porphyry coppers* the alteration and mineralization processes are of such a nature that they create favorable conditions for block caving methods, both in the ore and the capping rocks. Close fracturing is a universal characteristic in this type of deposit and also in the asbestos, iron, and limestone mines studied.

Summary of Geologic Factors

Numerous basic facts in 54 different cases that were studied concerning the relation of geological data to mining methods may be summarized under three general headings: 1—planes of weakness. 2—effects of mineralization, 3—other characteristics.

Planes of Weakness

1—Open stopes with pillars are generally used in deposits notable for their simplicity of structure, absence of extensive faulting and alteration, and competency of rocks and ore at relatively shallow depths.

2—Bedding planes may or may not be important causes of rock failure or weakness, depending upon their spacing, nature of partings, and type of rock.

3—Complexity of structure may or may not contribute to weakening of ore and rocks, depending on the nature of contacts and component members.

4—Rock weakness may be caused by either pre-ore or post-ore faulting, the latter being the more common cause. Pre-ore fractures may be healed by mineralization or serve as a means of weakening by alteration or subsequent movement.

5—Schistosity, particularly in relatively fine-grained rocks, usually is not a serious source of weakness.

6—Even slight post-mineral movement may cause failure in a brittle ore complex.

7—Contacts between ore and wall rocks often form important planes of weakness.

8—Intersections of shear zones may cause prominent irregularities that create mining problems.

9—The presence of joints, faults, contacts, and bedding planes relieves stresses that would otherwise be built up in the rock.

10—Internal *gouge slips* in the ore affect its strength and consequently the method of mining.

Effects of Mineralization

1—The conversion of limestone to chert and flint in the Tri-State district strengthened the rocks.

2—At the Horne mine, Noranda, Que., and at Ducktown, Tenn., the massive sulphide ores are very strong. Microscopic studies of sulphide ore show that

Table II. Intensity Factors for Non-Caving Methods

Stoping Method	Pre-Mineral Faulting, Etc.	Post-Mineral Faulting, Etc.	Alteration	Silicification or Strengthening Mineral
Open stopes	Negligible to moderately strong	Negligible to moderately weak	Negligible to moderately weak	Negligible to moderately strong
Sublevel	Negligible to strong	Negligible to moderately weak	Negligible to moderately weak	Negligible to strong
Shrinkage	Moderately weak to strong	Negligible to moderately weak	Negligible to moderately strong	Negligible to very intense
Cut-and-fill	Moderately strong to very intense	Moderately strong to very intense	Moderately strong to very intense	Negligible to very intense
Square-set	Moderately strong to very intense	Moderately strong to very intense	Moderately strong to very intense	Negligible to very intense

even very small cracks are healed by mineralization, which contributes materially to the strength of ore.

3—Silicification and mineralization of rhyolite breccias has in many cases strengthened them sufficiently so that they will support themselves in large openings.

4—The strengthening effect of silicification apparently may in some cases more than offset the weakening effects of sericitization. A high silica content of the original rocks is often essential for silicification to take place.

5—Uniformity of grade of ore affects the choice of mining methods: uniform grades usually require simpler methods of mining.

Other Characteristics

1—Strong rocks are found among igneous, sedimentary, and metamorphic type. Shale is almost universally weak.

2—A study of the Hollinger deposit shows that a majority of the geologic events in its history had an important effect upon the final structural strength of the ore and rock.

3—Branching of orebodies usually requires the use of a selective or closely supporting method of mining.

4—At the United Verde mine the rocks are strong to weak in the following order: massive sulphide (non-ore), porphyry, and schist.

5—In some cases, for otherwise similar structural conditions, the size of veins was the determining factor in a choice between shrinkage stoping and cut-and-fill, the first being employed in wider veins.

6—Where the mineralization has tended to follow joints, planes, and fractures into the walls of replacement veins, close wall support is necessary for complete extraction and clean mining.

7—Alteration of rocks in the Butte area was influenced by temperature of the solutions, chemical composition, and concentration. Degree of alteration is apparently not entirely dependent upon the degree of crushing or fracturing of the rock.

8—The persistence or continuity of orebodies both horizontally and vertically seriously affects the method of mining. Discontinuous deposits usually require selective methods.

9—In certain cases slates and graphitic rocks may act as lubricants along slip planes and facilitate adjustments of rock pressures and stresses.

10—Hard brittle rocks are more susceptible to bursting than soft weak ones.

11—The factors influencing the incidence and severity of rockbursts are: depth of workings, structural features of ore and enclosing rocks, dip of orebody, concentration of mining operations, and rate of mining.

12—In deposits employing caving methods a weak capping is usually an important requisite. Weakness is generally due to fracturing, alteration, leaching, or the presence of inherently weak rocks.

13—Drying of mine rock by pumping or ventilation often causes certain types of rocks to slack, slough, or crumble.

14—Massive size of an ore deposit is generally, but not always, a necessary requisite to successful block caving.

15—Variation in the type of wall rock or in the degree of faulting or alteration may permit the use of different types of mining in the same deposit.

Conclusions

Of all of the causes of weak mine rock structures, post-mineral faulting and *destructive* alteration are the most important. Pre-mineral faulting usually results in the weakening of rocks and ores, but to a lesser degree than the first two causes, usually because the mineralization processes healed the fractures caused by previous dynamic movement. Both silicification and certain other types of mineralization have a strengthening effect upon rock structures. They may come about as a result of either primary or secondary processes.

There appears to be no direct correlation between genetic rock types and their tendency toward being strong or weak. Their strength is a function not only of the physical properties of the rocks themselves, but their environment and geological history as well.

An interesting comparison can be drawn between the four factors given in Table II.

As might be expected, there is considerable overlapping of the effects of the more crucial geological factors influencing strength of rock and ore. Effects of one factor may be offset by those of another. For example, silicification may create a very strong but brittle rock structure easily weakened by further dynamic movement of the rocks. This appears to be true for some of the square-set mines studied.

Application of geological data to mining methods must be combined with other scientific methods, and if the largely qualitative geological data that have been described are to be employed to best advantage, they must be used in conjunction with available quantitative data concerning stress analysis and the physical properties of rock specimens of laboratory size.

References

[1] H. E. McKinistry, geologist, Harvard University: Personal communication.

[2] C. F. Jackson and E. D. Gardner: Stoping Methods and Costs. U. S. Bur. Mines *Bull. 390* (1936).

[3] R. U. King: A Study of Geologic Structure at Climax in Relation to Mining and Block Caving. *Trans.* AIME (1946) **163**.

Characteristics of Titaniferous Concentrates

Study of Chemical and Mineralogical Composition of Beach Sands from North Carolina, Florida, Brazil, and India Leads to a Theory Concerning the Origin of Titaniferous Concentrates . . .

Since the composition and structure of the beach sand concentrates correspond so closely to what would be expected of ilmenites that have been altered by oxidation and leaching, there is no reason to postulate the existence of the hypothetical compound, arizonite, to account for the composition of titaniferous beach sand concentrates. The four concentrates studied are examples of the different products that can result from various degrees of alteration of primary ilmenite.

by L. E. Lynd, H. Sigurdson, C. H. North, and W. W. Anderson

CONSIDERABLE uncertainty is revealed in the literature regarding the nature of the titanium minerals which make up the bulk of the heavy, opaque fractions of numerous beach sand deposits of the world. An investigation was made, therefore, of beach sand concentrates of North Carolina, Florida, Brazil, and India. Magnetic concentration, X-ray, and microscope data were obtained which show that these concentrates consist essentially of ilmenite and its alteration products. The alteration, brought about by oxidation and leaching of iron values, has resulted in upgrading the ores to a range of 56 to 64 pct TiO_2, as compared with 52.7 pct in ilmenite of theoretical composition.

L. E. LYND, Student Associate AIME, C. H. NORTH, and W. W. ANDERSON are with the Research Department, Titanium Division, National Lead Co., South Amboy, N. J. H. SIGURDSON is associated with P. R. Mallory Co., Indianapolis.

Discussion on this paper, TP 3815H, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, Oct. 14, 1953. New York Meeting, February 1954.

Conclusions drawn regarding the nature of the dominant titanium-bearing mineral in beach sand deposits have been based mainly on chemical composition, specific gravity, and petrography. This material has usually been referred to as ilmenite or weathered ilmenite. It has also been referred to as *arizonite* because in chemical composition it resembles an occurrence in Arizona which was described by Palmer[1] in 1909.

The chemical composition of Palmer's arizonite corresponded closely to the formula $Fe_2O_3 \cdot 3TiO_2$. From approximate measurements of crystal angles on a large imperfect crystal with rounded faces, the

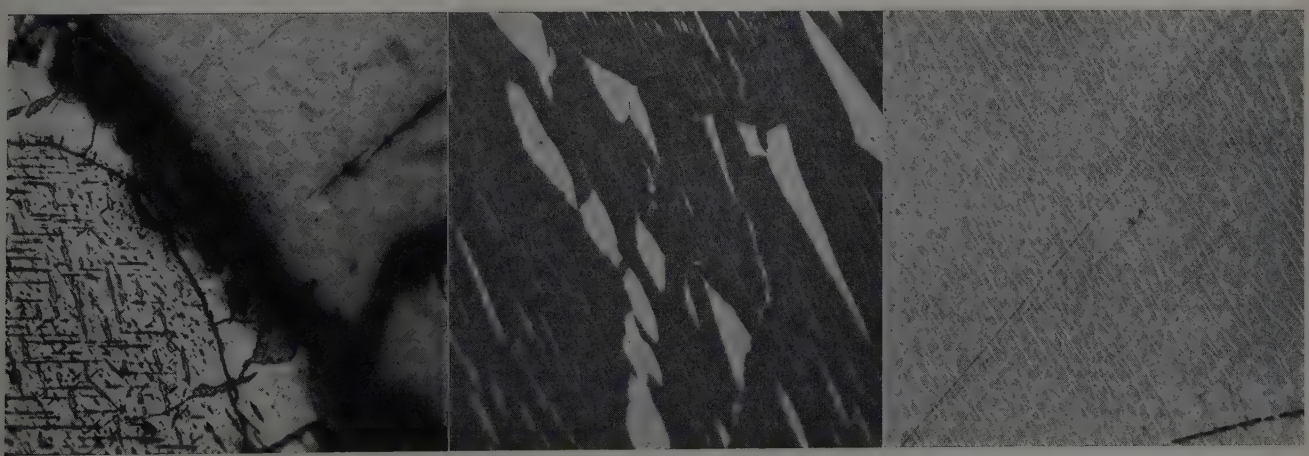


Fig. 1 (left)—MacIntyre rod mill feed showing coarse homogeneous ilmenite and coarse magnetite containing intergrown ilmenite. Gangue minerals rim ilmenite bodies in magnetite and often fill intergranular space. X700. Fig. 2 (center)—Ore from Baie St. Paul showing oriented exsolution lenses of hematite (light) in coarse ilmenite (dark). Dark streaks within the hematite are exsolution bodies of ilmenite. X700. Fig. 3 (right)—Skondal ore, showing very fine exsolution lenses of hematite (light gray) in ilmenite (dark gray). X700.

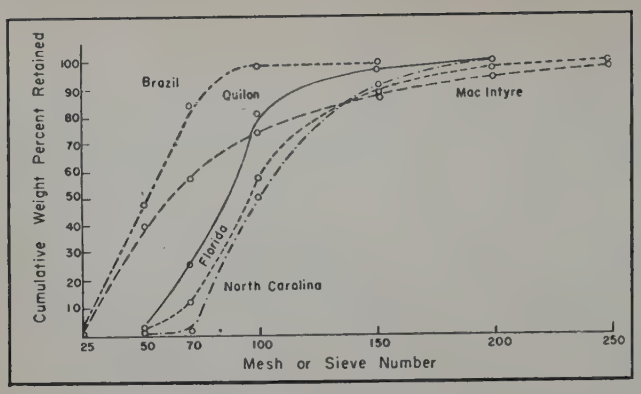


Fig. 4—Results of sieve analysis of ores.

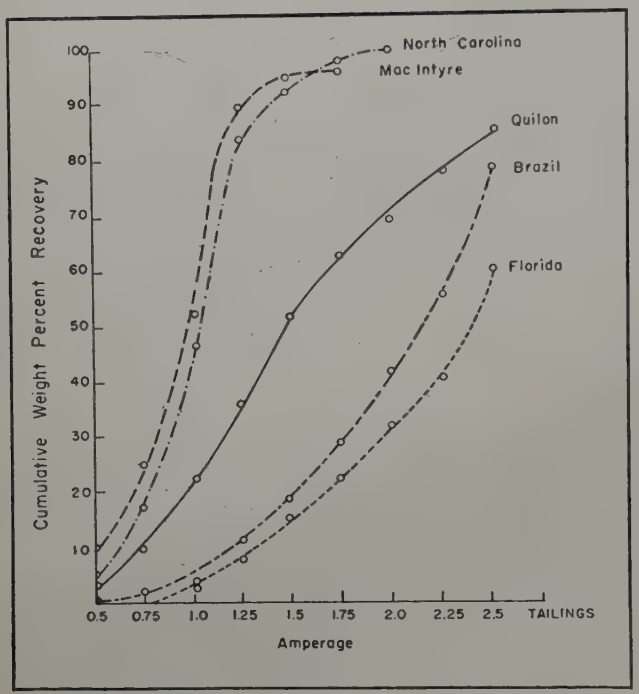


Fig. 5—Cumulative weight percent of ores concentrated at various amperages.

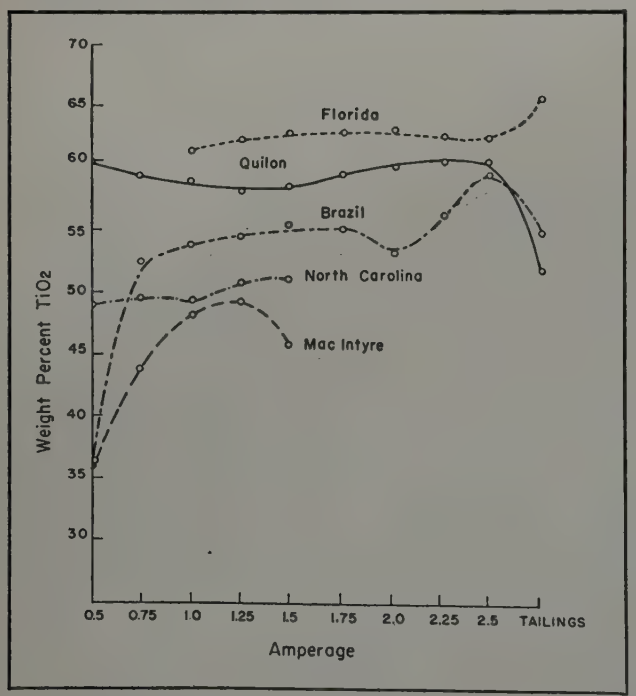


Fig. 6—TiO_2 contents of ore fractions concentrated at various amperages.

crystal form was thought to be monoclinic, not corresponding to any mineral then known. However, the possibility of another symmetry was admitted, and in 1944 Ernst[2] showed that considering the accuracy of measurement the symmetry can be interpreted with equal justification as rhombohedral, corresponding to that of hematite. After making studies by X-rays and polished sections, Ernst concluded that arizonite is an example of extensive replacement of specular hematite by rutile.

In 1950 Overholt, Vaux, and Rodda[3] concluded on a basis of X-ray measurement that arizonite is weathered ilmenite. They also pointed out that with vertical polarized light alteration of ilmenite can be observed microscopically in polished sections.

Attempts by Pesce[4] and later by Ernst[2] to synthesize a compound $Fe_2O_3 \cdot 3TiO_2$ were unsuccessful. With mixtures of this composition Ernst obtained only pseudobrookite ($Fe_2O_3 \cdot TiO_2$) and small amounts of rutile, indicating extensive solid solution of TiO_2 in pseudobrookite.

Although evidence is lacking for the existence of a compound $Fe_2O_3 \cdot 3TiO_2$, the name *arizonite* has been mentioned frequently as applicable to large beach sand deposits of titanium minerals.

In 1945 Miller[5] stated that the so-called ilmenite of Travancore is arizonite. In 1949 Thoenen and Warne[6] listed arizonite as a minor constituent of ilmenite-bearing beach sands. Gillson[7,8] applied the name to the ilmenite deposits of Florida; Travancore, India; and some parts of Brazil, stating, however, that the mineral had not been proved to be truly arizonite.

A number of investigators who have studied Florida sands have favored the view that the material in question is actually weathered ilmenite. On X-ray examination of Florida ore in 1945, Miller[5] found weak lines of ilmenite and concluded that the ore was unlike the type of arizonite from Arizona. In 1948 Creitz and McVay[9] and Spencer,[10] using X-ray and other evidence, concluded that the heavy opaque minerals from Florida sands are leucoxenes having variable TiO_2 contents and that they may have formed by weathering of the ilmenite originally present in the deposit.

Cannon[11] (1950) believed: 1—that the Florida deposits are altered ilmenites, 2—that the loss of iron takes place molecule by molecule throughout the series from true ilmenite to pure leucoxene, and 3—that pure leucoxene is a mass of microfine rutile crystals. No X-ray data on Quilon or Brazil sands were found in the literature.

No systematic investigation of polished sections of *ilmenite* sands in reflected light has been described so far, and no micrographs have been presented to give evidence of structure. Creitz and McVay[9] examined polished sections but stated that these did not provide useful information.

Review of Ilmenite Mineralogical Compositions

To provide a suitable background against which titaniferous beach sands can be considered, a few typical associations of ilmenite in various rock deposits are described below.

By far the greater number of ilmenite rock deposits in the world are intimately associated with iron oxide that may be present as hematite or magnetite. Depending on conditions during crystallization, the ilmenite and iron oxides may form coarsely granular aggregates, fine intergrowths, or solid solutions.

Table I. Screen Analyses of Head Samples of Ores, Cumulative Weight Percent Retained

Sieve No.	Sieve Opening, In.	MacIntyre Ilmenite	Brazilian Sand	North Carolina Sand	Quilon Sand	Florida Sand
25	0.028	0.7	2.2	0.0	0.0	0.0
50	0.0117	39.2	48.8	0.75	2.2	2.0
70	0.078	57.2	84.0	1.5	26.4	11.0
100	0.0055	74.5	98.5	51.0	81.3	58.0
150	0.0041	85.5	100.0	92.0	96.8	89.0
200	0.0029	94.5		100.0	99.0	98.0
250	0.0024	97.5			99.5	99.5
325	0.0017	98.5			100.0	100.0
Ore grain characteristics		Angular	Partially rounded	Well rounded	Well rounded	Well rounded

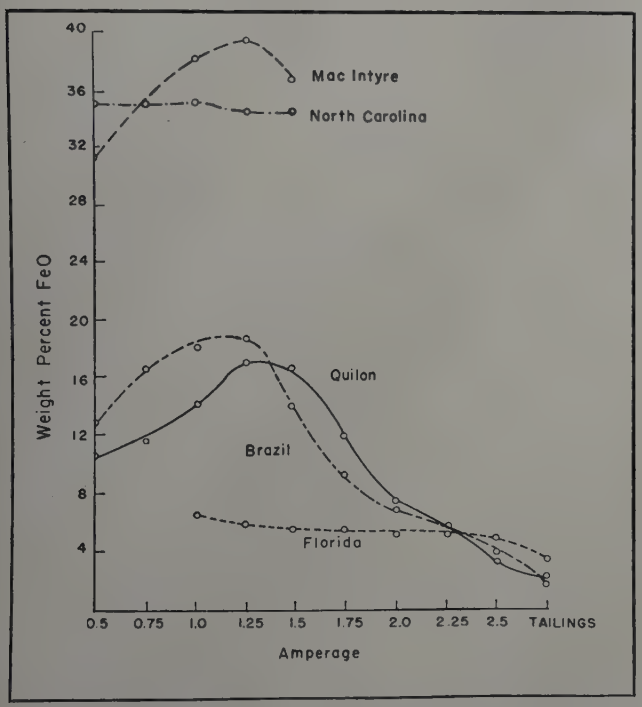


Fig. 7—FeO contents of ore fractions concentrated at various amperages.

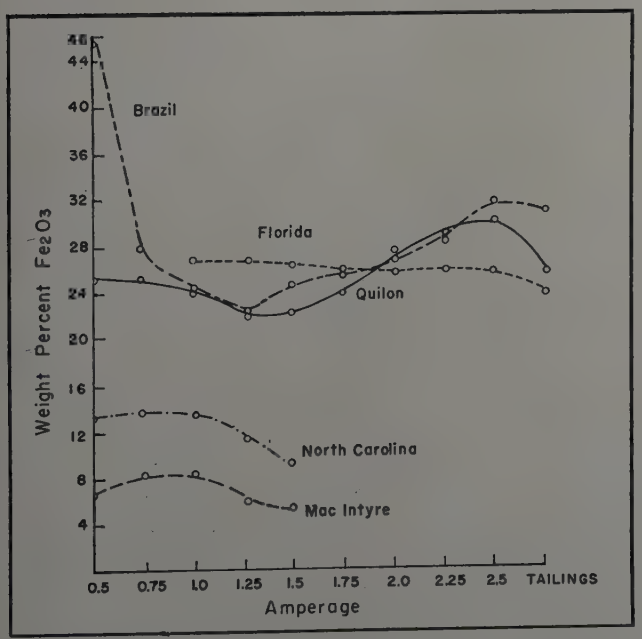


Fig. 8—Fe_2O_3 contents of ore fractions concentrated at various amperages.

In the MacIntyre orebody at Tahawus, N. Y., ilmenite and magnetite are granular. While the ilmenite fraction is free from visible magnetite at X1350, the magnetite contains lamellae of ilmenite. This is illustrated in Fig. 1.

In the deposits at Baie St. Paul and Allard Lake, Quebec, hematite is present as elongated lenses in parallel orientation in the ilmenite matrix, see Fig. 2. In the orebody at Skondal, Norway, hematite is present as uniformly disseminated fine lenses in the ilmenite matrix, Fig. 3.

On examination of polished sections in reflected light with a Bausch and Lomb research metallograph the characteristic structural features of each of the above unaltered ore types are readily discernible. The presence of similar textural features in grains from beach sand deposits suggests that primary ilmenite was the source of the deposits.

Microscopic and X-ray examination of ilmenites in this laboratory during the period 1947 through 1950 had given specific evidence of variations in structural and mineralogical compositions from ore to ore and within individual ores. In the case of beach sands the variations were apparently caused by different degrees of alteration of each ore and of its individual grains.

Table II. Chemical Composition of Various Ilmenite Concentrates

Type of Ore	TiO_2, Pct	FeO, Pct	Fe_2O_3, Pct	Other
MacIntyre	44.6	36.2	6.9	12.3
North Carolina	49.0	33.9	12.2	4.9
Quilon	58.2	10.2	24.3	7.3
Brazil	56.1	7.0	27.7	9.2
Florida	64.1	4.7	25.6	5.6
Theoretical ilmenite	52.7	47.3		

To study these variations more effectively and to correlate the degree of alteration with other features such as chemical and mineralogical composition, it was considered advisable to separate each ore into fractions which would show varying degrees of alteration. Magnetic concentration was selected as the most effective way of achieving such a separation. Evidence of wide variations in the composition of different magnetic fractions of beach sand ilmenites from Senegal, Travancore, and Florida had been observed previously by members of this organization.

Description of Ores

The ores selected for investigation differed markedly in their degree of alteration. North Carolina beach sand was only slightly altered, while sands of Quilon, India; Guarapari, Brazil; and Trail Ridge, Fla. showed increasing degrees of alteration. The Florida ore fraction used was that designated as the magnetic concentrate and did not include the most highly weathered portion of the ore, usually referred to as leucoxene, which is grayish white in color, insoluble in acid, and 80 to 90 pct TiO_2. MacIntyre ilmenite was selected as a standard unaltered ilmenite because of its virtual freedom from iron oxide intergrowths.

Ores selected for testing differed in particle shape. Ore grains from New York State were highly angular, grains from Brazil angular but partially rounded, and grains from Florida, North Carolina, and Quilon well rounded.

Screen analyses, Table I and Fig. 4, on representative head samples of these ores show that all were

Table III. Concentration, Chemical, and Mineralogical Data on Concentrates of Various Titaniferous Ores

Amp.	Prod. Dist., Wt Pct	Analyses-Weight, Pct				Recovery						Weight Ratio		Mineralogical Composition, Total Ore Fraction, Pct		
						TiO_2		FeO		Fe_2O_3		Fe(Total)	Fe^{3+}			
		TiO_2	FeO	Fe_2O_3	Others (Gangue)	Wt	Wt Pct	Wt	Wt Pct	Wt	Wt Pct	TiO_2	Fe(Total)	Fresh Ilmenite	Altered Ilmenite	Others
MacIntyre																
0.5	9.5	36.2	31.5	6.9	25.4	3.4	7.6	3.0	8.3	0.6	8.7	0.81	0.17	95+	0	<5 magnetite
0.75	15.5	44.0	35.0	8.3	12.7	6.8	15.3	5.4	14.9	1.3	18.8	0.75	0.18	95+	0	Trace magnetite
1.0	27.3	48.4	38.3	8.4	4.9	13.2	29.6	10.5	29.0	2.3	33.3	0.74	0.17	100	0	
1.25	37.3	49.6	39.6	5.4	5.4	18.5	41.5	14.8	40.9	2.0	29.0	0.70	0.11	100	0	
1.50	5.2	45.9	36.8	4.7	12.6	2.4	5.4	1.9	5.3	0.2	2.9	0.70	0.10	100	0	
1.75	0.9	19.9	22.7	8.9	48.5	0.2	0.4	0.2	0.5	0.1	1.5	1.2	0.26	100	0	
Tailing	4.3	2.2	10.6	9.3	77.9	0.1	0.2	0.4	1.1	0.4	5.8	6.7	0.44			
Total	100.0					44.6	100.0	36.2	100.0	6.9	100.0					
North Carolina																
0.5	5.0	49.0	35.1	13.0	2.9	2.4	4.9	1.7	5.0	0.6	4.9	0.74	0.25	90	5	5 magnetite
0.75	12.0	49.5	35.1	13.3	2.1	5.9	12.0	4.2	12.4	1.6	13.1	0.74	0.25	95	<5	Trace magnetite
1.0	30.0	49.4	35.5	13.1	2.0	14.8	30.2	10.6	31.3	3.9	32.0	0.74	0.25	90	5	5 magnetite
1.25	36.7	50.8	34.5	11.4	3.3	18.6	38.0	12.7	37.5	4.2	34.4	0.69	0.23	90	5	5 magnetite
1.50	8.3	51.3	34.4	8.9	5.4	4.3	8.8	2.8	8.5	0.7	5.7	0.64	0.19	90	8	2 magnetite
1.75	5.9	42.9	24.7	14.6	17.8	2.5	5.1	1.5	4.4	0.9	7.4	0.68	0.35	80 to 85	15	<5 magnetite
2.0	2.1	22.4	12.8	13.0	51.8	0.5	1.0	0.3	0.9	0.3	2.5	0.85	0.48	20	80	
Total	100.0					49.0	100.0	33.9	100.0	12.2	100.0					
Quilon																
0.5	2.9	60.5	10.9	25.3	3.3	1.8	3.1	0.3	2.9	0.7	2.8	0.43	0.68	20	80	
0.75	6.5	59.5	11.7	25.3	3.5	3.9	6.7	0.7	6.9	1.6	6.4	0.45	0.66	25	75	
1.0	12.3	59.1	13.5	24.2	3.2	7.3	12.6	1.7	16.7	3.0	12.1	0.46	0.62	30	70	
1.25	13.1	58.3	17.1	21.5	3.1	7.6	13.1	2.2	21.6	2.8	11.3	0.49	0.53	35	65	
1.50	15.5	58.7	16.3	21.7	3.3	9.1	15.6	2.5	24.5	3.4	13.7	0.47	0.54	35	65	
1.75	10.1	59.7	12.1	24.2	4.0	6.0	10.3	1.2	11.8	2.4	9.7	0.44	0.66	20	80	
2.0	8.3	60.1	7.8	27.2	4.9	5.0	8.6	0.6	5.9	2.3	9.3	0.42	0.76	10	90	
2.25	7.9	60.8	5.6	28.8	4.8	4.8	8.2	0.4	3.9	2.3	9.3	0.40	0.82	5	95	
2.5	7.0	60.7	4.1	29.5	5.7	4.2	7.2	0.3	2.9	2.1	8.5	0.39	0.86	2	98	
Tailing	16.4	52.0	2.2	25.7	20.1	8.5	14.6	0.3	2.9	4.2	16.9	0.38	0.91	0	100	
Total	100.0					58.2	100.0	10.2	100.0	24.3	100.0					
Brazil																
0.5	0.5	30.4	12.6	45.5	5.5	0.2	0.3	0.1	1.4	0.2	0.7	1.1	0.71	30	50	20 magnetite
0.75	0.9	52.4	16.3	28.0	3.3	0.5	0.9	0.1	1.4	0.2	0.7	0.62	0.61	40	60	Trace magnetite
1.0	2.4	54.0	17.9	24.3	3.8	1.3	2.3	0.4	5.7	0.6	2.1	0.57	0.55	50	50	Trace magnetite
1.25	7.5	54.6	18.6	21.7	5.1	4.1	7.3	1.4	20.0	1.6	5.8	0.54	0.51	40	60	
1.50	7.6	55.6	13.4	24.6	6.4	4.2	7.5	1.0	14.3	1.9	6.9	0.50	0.62	25	75	
1.75	10.1	55.3	9.2	26.2	9.3	5.6	9.9	0.9	12.9	2.6	9.4	0.46	0.72	15	85	
2.0	12.3	53.3	7.4	26.9	12.4	6.6	11.8	0.9	12.9	3.3	11.9	0.46	0.77	10	90	
2.25	14.9	56.4	5.6	28.4	9.6	8.4	15.0	0.8	11.4	4.2	15.2	0.43	0.82	1	99	
2.5	22.0	59.7	3.5	30.7	6.1	13.2	23.6	0.8	11.4	6.7	24.2	0.41	0.89	0	100	
Tailing	21.8	55.1	2.9	29.4	12.6	12.0	21.4	0.6	8.6	6.4	23.1	0.41	0.90	0	100	
Total	100.0					56.1	100.0	7.0	100.0	27.0	100.0					
Florida																
1.0	3.5	61.4	6.5	27.0	5.1	2.1	3.3	0.2	4.3	0.9	3.5	0.39	0.77	5	95	
1.25	4.7	62.6	5.6	27.0	4.8	2.9	4.5	0.3	6.4	1.3	5.1	0.37	0.81	2	98	
1.50	6.9	63.0	5.5	26.6	4.9	4.3	6.7	0.4	8.5	1.8	7.0	0.36	0.81	2	98	
1.75	7.7	63.0	5.4	26.4	5.2	4.8	7.5	0.4	8.5	2.0	7.8	0.36	0.82	2	98	
2.0	8.5	63.4	5.1	26.3	5.2	5.4	8.4	0.4	8.5	2.2	8.6	0.35	0.82	1	99	
2.25	9.3	63.2	5.3	26.6	4.9	5.9	9.2	0.5	10.6	2.5	9.8	0.36	0.82	0	100	
2.5	19.3	63.1	5.0	26.3	5.6	12.2	19.0	1.0	21.3	5.1	19.9	0.35	0.82	0	100	
Tailing	40.1	66.1	3.8	24.4	5.7	26.5	41.4	1.5	31.9	9.8	38.3	0.30	0.85	0	100	
Total	100.0					64.1	100.0	4.7	100.0	25.6	100.0					
Theoretical ilmenite		52.7	47.3									0.70	0.00			

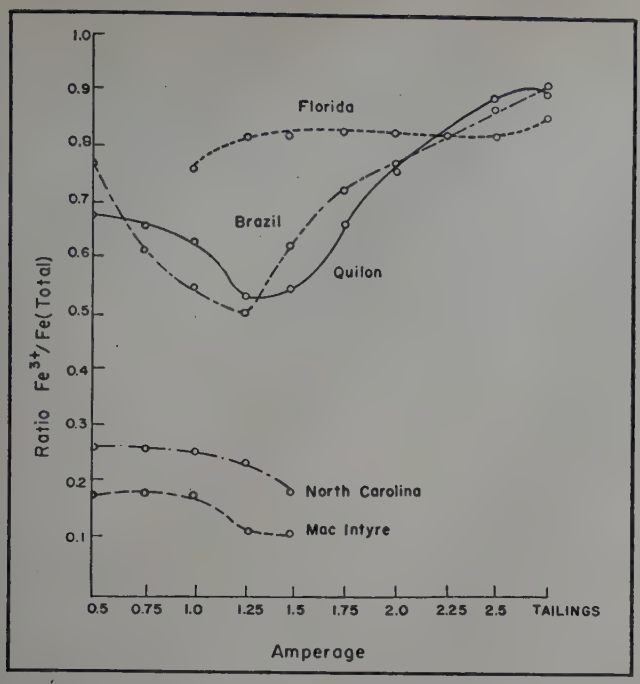


Fig. 9—Ratio Fe^{3+}/Fe(total), indicating relative degree of oxidation in ore fractions concentrated at various amperages.

essentially finer than 25 mesh and 85 to 100 pct were retained on 150 mesh. Chemical analyses of the ores, presented in Table II, show that the TiO_2 contents ranged from 44.6 to 64.1 pct, the FeO from 4.7 to 36.2 pct, and the Fe_2O_3 from 6.9 to 28.2 pct.

Methods and Results of Investigation

Magnetic Concentration: The ores were separated into fractions in order of decreasing magnetic susceptibility by a Stearns ring type *D* magnetic separator, No. 0, a two-pole machine rated at 28,000 ampere-turns and 440 maximum wattage. The separator was operated at 110 v in conjunction with a rheostat which allowed variation of the current from 0.5 to 2.5 amp. The maximum air-gap setting of 3/16 in. was maintained throughout the tests. A Syntron vibratory feeder machine, Model FM-0-10, provided a satisfactory means of feed control.

A representative sample of each ore was separated into several fractions by the following method:

1—An initial concentrate was recovered at the minimum setting of 0.5 amp.

2—The tailings fraction from this step was reprocessed to yield a concentrate at 0.75 amp.

3—This procedure was repeated at successively higher amperages, as indicated in Table III, until all the ore was concentrated or the maximum amperage of 2.5 was reached. The weight percent distributions of the fractions obtained from each ore at the various amperages are presented in Table III and Fig. 5.

Differences in magnetic susceptibility of the various ores were pronounced. MacIntyre and North Carolina, although differing considerably in particle size range and shape, were almost identical in magnetic behavior. Both ores required only 1.5 to 1.75 amp for recovery of practically all the titanium values in the concentrates, whereas with ore from Quilon and Brazil, and especially with ore from Florida, considerable percentages of the titanium values remained in the tailings from the 2.5-amp concentration. Although ores from Brazil and Florida differed considerably in particle size and shape their magnetic behavior was similar. Quilon ore resembled Florida ore with respect to particle size but was considerably more magnetic. It was therefore apparent that particle size differences between the ores did not affect significantly their magnetic behavior or the distribution of the ore concentrates.

No doubt further separation of the 2.5-amp tailings from ores of Quilon, Brazil, and Florida could be achieved readily by the use of higher intensity separation equipment.

Chemical Analysis: Each of the products obtained from the magnetic concentrator was analyzed for TiO_2, FeO, and Fe_2O_3 by standard analytical procedures. Results of these analyses, indications of the amount of gangue constituents present in each fraction, and weight and weight percent distributions of TiO_2, FeO, and Fe_2O_3 are listed in Table III. The chemical data are also expressed graphically in Figs.

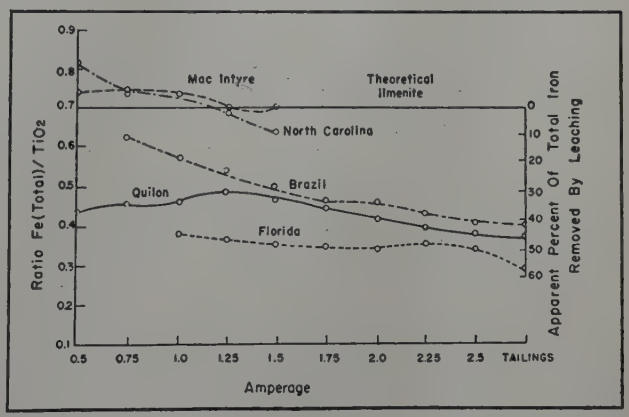


Fig. 10—Ratio Fe(total)/TiO_2, indicating relative degree of leaching of iron oxides from ore fractions concentrated at various amperages.

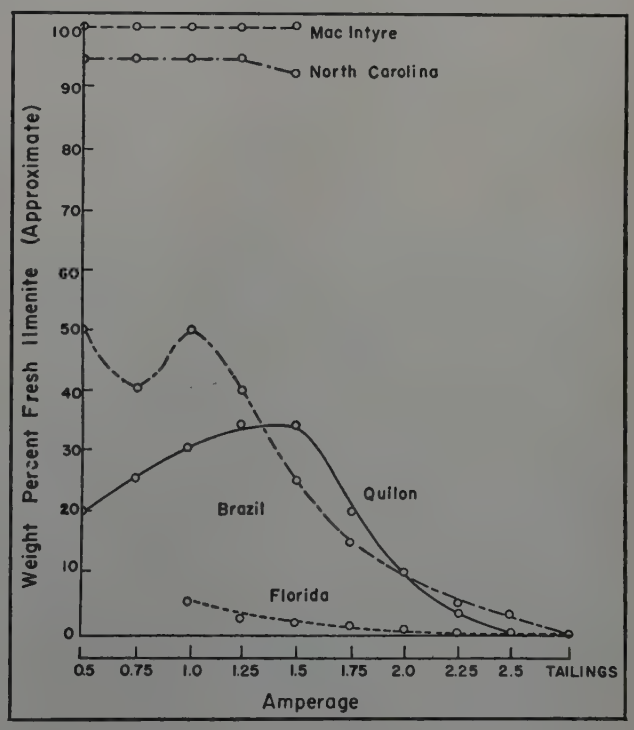


Fig. 11—Fresh ilmenite contents of ore fractions concentrated at various amperages.

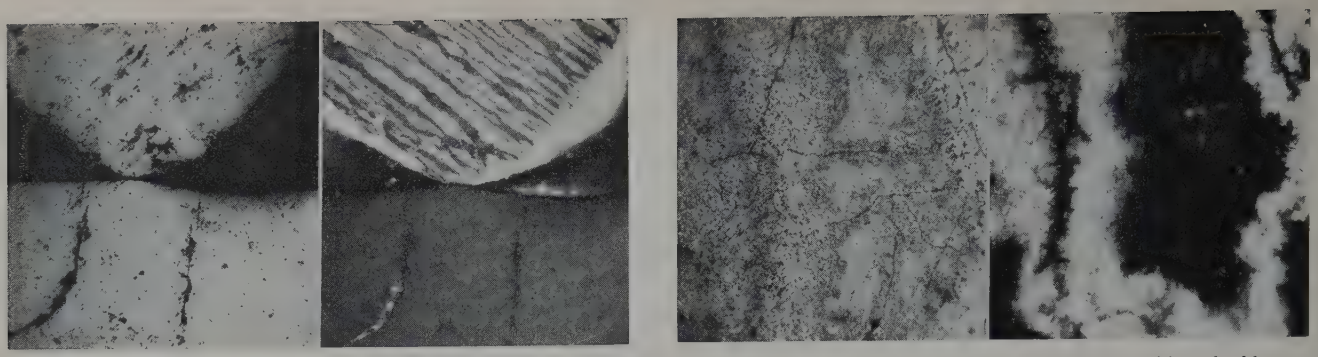


Fig. 12 (left)—Brazilian ore grains, altered to different degrees, in bright light (left) and in polarized light (right). More highly altered areas are brighter in polarized light than are less altered areas. Originally X250; reduced one-half. Fig. 14 (right)—Highly altered grain of Quilon ore, in bright light (left) and in polarized light (right). The most highly altered portions are finely granular, porous, and bright in polarized light. Alteration appears to have proceeded most rapidly along fractures. Originally X1000; reduced one-half.

6, 7, and 8. Some of the values plotted for tailings fractions are erratic because of high gangue content.

Calculated ratios of Fe^{3+}/Fe(total) for each fraction are presented in Table III and Fig. 9. These ratios range in general from 0.10 to 0.25 for MacIntyre and North Carolina ores, from 0.53 to 0.91 for Quilon and Brazilian ores, and from 0.77 to 0.85 for ore from Florida, in contrast to a ratio of 0.0 in theoretical ilmenite. The values give an indication of the degree of oxidation of the different fractions and vary considerably from ore to ore.

Calculated ratios of Fe(total)/TiO_2 for each fraction range in general from 0.64 to 0.81 for MacIntyre and North Carolina ores and from 0.30 to 0.62 for ores of Quilon, Brazil, and Florida, compared to a ratio of 0.70 for theoretical ilmenite. These values, which are shown in Table III and Fig. 10, give an indication of the degree of leaching of iron from the various fractions.

Microscopic Examination: A sample of each fraction of the various ores was mounted in plastic, and polished sections were prepared by grinding with coarse and fine carborundum on a cast iron lap, followed by polishing on billiard cloth with a slurry of Linde A alumina. Buehler laboratory polishing equipment was used.

The polished sections were examined with a Bausch and Lomb research metallograph at high magnification (X1350). It was found that lower magnifications did not bring out distinctly the differences in color between fresh ilmenite and slightly altered ilmenite. The use of a high-power oil immersion objective, however, brought out color differences in good contrast, both in bright and polarized light, so that fresh ilmenite could be distinguished readily from even slightly altered material. Since it was considered of particular interest to note the relative amounts of fresh ilmenite in the various fractions, estimates of these amounts are included in Table III under mineralogical composition and are presented graphically in Fig. 11. These estimates are considerably lower than the percentage of grains containing fresh ilmenite, since the amount of fresh ilmenite in the various grains ranged from 0 to 100 pct. The overall fresh ilmenite contents of the head samples of ore amounted to 100 pct for MacIntyre, 90 pct for North Carolina, 20 pct for Quilon, 10 pct for Brazil, and 1 pct for Florida.

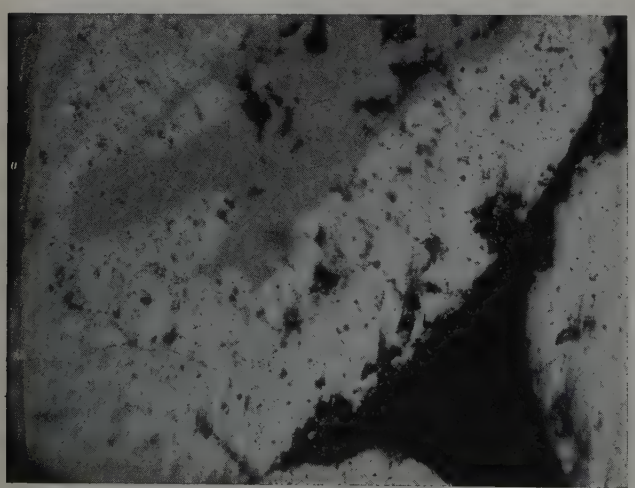


Fig. 13—Quilon ore grain, partially altered, with fresh ilmenite core (dark gray), surrounded by light gray granular alteration products. X1000.

The material designated as fresh ilmenite was brown to pinkish brown in bright light in contrast to the grayish-white, minutely granular, porous appearance of the altered portions. The fresh ilmenite was present both as discrete grains and as skeletal remnants in partially altered grains. Frequently the fresh homogeneous ilmenite graded into gray altered material.

The appearance of the altered ilmenite varied, depending on the degree of alteration. Some grains appearing grayish white and granular in bright light were dark in polarized light, as was fresh ilmenite. Other grains showed dark brown to bright yellow reflections in polarized light, in a wide variety of patterns: light and dark alternating bands, mottled areas, diffuse brown grains of various tones, or rims of light alteration products around a core of fresh or less altered material. The micrographs of polished sections of typical altered grains are shown here, see Figs. 12-16.

X-ray Diffraction: Powder X-ray diffraction patterns were obtained for selected ore fractions with a Philips high-angle Geiger-counter spectrometer with Zr-filtered Mo radiation. Samples were ground to well below 200 mesh and packed dry into an aluminum specimen holder so that a plane rectangular area was presented to the X-ray beam. The geometry of the spectrometer was set for optimum resolution and intensity of response. Patterns were recorded at a scanning rate of ¼° per min over an angular range corresponding to d-spacings from about 1.00 to 4.00 A.

Table IV lists the mineral components identified in selected ore fractions. Patterns were compared directly with those of cryptocrystalline hematite, rutile from Florida, and MacIntyre ilmenite which in turn corresponded to ASTM card index data within the limit of experimental errors.

Table IV. A Summary of Mineral Composition of Magnetic Concentrates Based on X-Ray Pattern Intensities, Interpreted from Data Provided by Chemistry, the Microscope, and the Electron Microscope

Description of Sample	Relative Amounts of Constituents*			
	Ilmenite	Hematite	Rutile	Unidentified
North Carolina ore				
1.25 amp concentrate	Very high	Trace	Absent	Absent
Quilon ore				
0.5	Major	Low	Low	Absent
1.25	Major	Minor	Absent	Absent
2.0	Minor	Minor	Minor	Trace
2.5	Low	Low	Major	Trace
Brazil ore				
1.0	Major	Low	Low	Absent
2.25	Low	Low	Minor	Trace
Florida ore				
1.0	Minor	Low	Major	Trace
2.5	Low	Low	Major	Trace

* Very high: accounts for nearly all of pattern strength.
* Major: accounts for 50 to 75 pct of pattern strength.
* Minor: accounts for 25 to 40 pct of pattern strength.
* Low: accounts for 10 to 20 pct of pattern strength.
* Trace: accounts for <10 pct of pattern strength.

Ore from North Carolina gave a strong pattern, indistinguishable from that of MacIntyre ilmenite. The other three beach sand ores (from Quilon, Brazil, and Florida) appeared mutually similar but with decreasing amounts of all crystalline components. The more highly magnetic fractions from Quilon and Brazil contained substantial amounts of normal ilmenite, but the more altered fractions appeared to contain more rutile and hematite with only negligible amounts of ilmenite. Both fractions of ore from Florida which were tested contained a small amount of ilmenite and a predominance of fine-crystallite rutile and hematite. It was almost impossible, from X-ray patterns alone, to distinguish whether the highly altered ore fractions were predominantly rutile or hematite. The patterns of the least magnetic ore fractions in this investigation often failed to contain any lines at 2.68 or 3.25 A, normally listed as the principal d-spacings of hematite and rutile respectively, but they exhibited broad weak maxima at d = 1.69 A and other spacings common to both. Therefore other factors, such as chemical composition, had to be considered in the interpretation of these patterns.

In determining crystallite diameters by X-ray diffraction it is customary to grind quartz or some other nominally pure mineral as fine as possible by mechanical means. Such a powder, assigned a crystallite size >1000 A., provides an *instrument width* or practical minimum diffraction line breadth. For the same instrument geometry, crystallites smaller than 1000 A. in diam exhibit weaker and broader lines than the standard pattern, in accordance with well-established formulae.[12,13] The smallest diameter that can be estimated in this way depends on the efficiency with which a broad line can be detected and on the crystallographic nature of the particular reflection plane. A practical minimum diameter might be about 50 A.

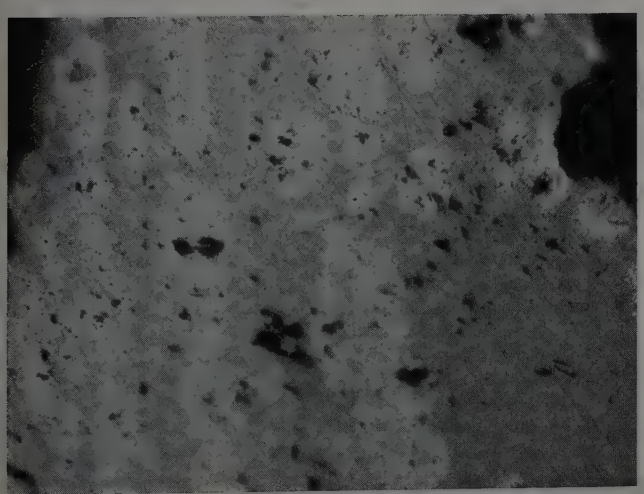
Table V lists peak intensities above background for several compounds tested under standard conditions of maximum response. Although not as rigorous a criterion as line breadth, intensity shows a regular progression downward as rutile or anatase TiO_2 crystallites decrease in size. A similar decrease in intensity is observed when two ores with relatively large crystallites are compared to a rouge hematite specimen.

Table V. Peak Intensity of Standard Patterns

Compound	Main Peak, d/n, A.	Intensity, Counts Per Sec
Rutile, sintered pigment*	3.25	138
Rutile, medium crystallite**	3.25	110
Anatase, fine crystallite†	3.54	42
Ilmenite, MacIntyre	2.75	160
Magnetite, MacIntyre	2.53	100
Hematite (polishing rouge)	2.68	50

* Diameter of (110) plane >1000 A.
** Diameter of (110) plane approximately 154 A.
† Diameter of (110) plane approximately 79 A.

When ore fractions from Quilon, Brazil, and Florida were run under the above conditions, their strongest pattern lines failed to equal the intensity of either the fine crystallite hematite or anatase, indicating that the predominant minerals in these ores approached a size range amorphous to X-rays, that is, crystallite diameters in the order of 50 to 100 A.



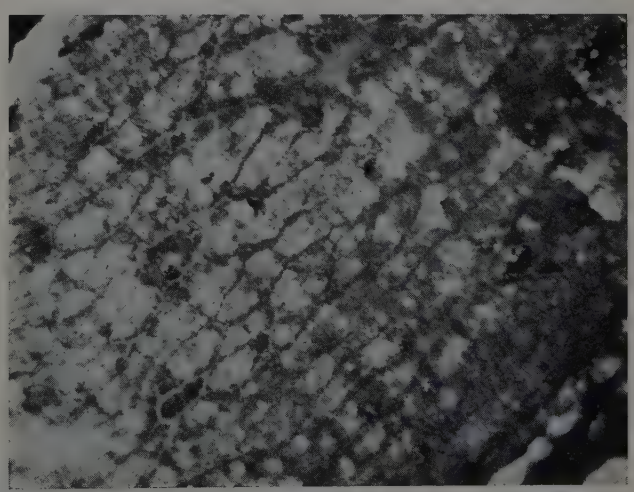


Fig. 15 (left)—Quilon ore grain in bright light, showing light gray altered areas and residual fresh ilmenite (dark gray). X1000.
Fig. 16 (right)—Highly altered Quilon ore grain in polarized light. Less altered material forms a network of gray stringers in highly altered material (light gray to white). X1000.

The factor of crystallite size helps to justify the apparent quantitative discrepancy between X-ray and microscopic estimates of ilmenite content in the ore fractions listed in Tables III and IV. X-ray estimates were based on the total pattern intensity of the detectable crystalline material rather than percent by weight of sample. Thus comparatively large crystallites of ilmenite in a few aggregates might indicate relatively high ilmenite content, whereas ultra-fine crystallites of alteration products in the remaining aggregates would go undetected.

A powdered specimen of arizonite obtained in 1946 from a small piece originating at Hockberry, Ariz., yielded a good pattern for a mixture of approximately 40 pct hematite, 40 pct anatase, and 20 pct finely crystalline rutile. This analysis agrees with the pattern described by Overholt, Vaux, and Rodda[3] and with the observations of Ernst.[2] Microscopic examination of this material revealed the presence of small hematite lenses in a matrix which was very similar in appearance to weathered ilmenite.

Electron Microscope Studies

Electron Diffraction: Patterns and micrographs were obtained of the 2.5-amp concentrate from the Florida ore and of three magnetic fractions of a less weathered Florida ore. Diffraction patterns of all samples indicated that rutile made up the bulk of the fine crystalline material present, ranging in basic particle size from about 50 to 1200A. The electron micrographs indicated that the aggregates were larger (0.1 to 1.5 microns) in the fractions of the less weathered ore than in the more weathered sample (0.005 to 0.8 microns).

Summary

The four beach sands investigated, although similar in general appearance and mode of occurrence, show considerable variation in composition and structure. The ore from North Carolina consists essentially of ilmenite, with minor amounts of intergrown magnetite, and is only slightly altered. In structure, composition, and magnetic behavior it is very similar to MacIntyre ilmenite occurring in Adirondack anorthosite and gabbro.

Examinations made by microscope, X-ray, and electron microscope of the various magnetic fractions obtained from the beach sands of Quilon, Brazil, and Florida show that ilmenite is present in various amounts in several fractions of each ore and that nearly all the non-ilmenite portion of the ores consists of a porous mixture of fine-grained rutile and iron oxide which formed as a result of alteration of the ilmenite.

Results of magnetic concentration tests emphasize the heterogeneous nature of the ores from Quilon, Brazil, and Florida. Over 90 pct of the MacIntyre and North Carolina ilmenite was recovered over a narrow amperage range on the magnetic separator, whereas ores from Quilon, Brazil, and Florida were recovered over a much wider and higher range.

The ores may be listed in order of increasing degree of alteration, and decreasing magnetic susceptibility, as follows: MacIntyre, North Carolina, Quilon, Brazil, Florida. Although more weathered than ore from Quilon, ore from Brazil is lower in TiO_2, since less of its ferric oxide has been removed by leaching, or since it contains highly weathered brown magnetite grains that are difficult to distinguish from brownish weathered ilmenite grains.

Quilon, Brazil, and Florida concentrates all contain ore grains ranging in composition from fresh ilmenite to a highly altered product approaching pure TiO_2 in composition. The more magnetic fractions of ores from Quilon and Brazil contain considerable amounts of unaltered ilmenite.

Florida ore, which is higher in TiO_2 content than the other ores, shows almost complete breakdown of the original ilmenite structure, its most magnetic fraction containing only about 5 pct of fresh ilmenite. Other samples of Florida ore have been encountered which exhibit a degree of alteration intermediate between that of the Florida ore studied and Quilon ore.

The ratios of Fe_2O_3 to total iron in the different ores indicate that oxidation has accompanied alteration. The higher TiO_2 to Fe ratio in the altered ores is good evidence that leaching of iron has occurred.

Since the composition and structure of the beach sand concentrates correspond so closely to what would be expected of ilmenites that have been altered by oxidation and leaching, there is no reason to postulate the existence of the hypothetical compound, arizonite, to account for the composition of titaniferous beach sand concentrates. The four concentrates studied are examples of the different products that can result from various degrees of alteration of primary ilmenite.

Acknowledgment

The authors express their appreciation to W. Lasko, who carried out the electron microscope studies, and to J. P. Wadington, who furnished the research samples of subpigmentary rutile and anatase.

References

[1] Chase Palmer: Arizonite, Ferric Metatitanate. *American Journal of Science,* Ser. 4 (1909) **28**, No. 166, pp. 353-356.

[2] T. Ernst: Fusion Equilibria in the System Fe_2O_3-FeO-TiO_2 and Notes on the Minerals Arizonite and Pseudobrookite. *Ztsch. Angew. Min.* (1943) **4**, pp. 394-409.

[3] J. L. Overholt, G. Vaux, and J. L. Rodda: The Nature of "Arizonite." *American Mineralogist* (1950) **35**, pp. 117-119.

[4] B. Pesce: *Concerning the Titanates of Iron. Gazz. chim. ital.* (1931) **61**, pp. 107-111.

[5] Roswell Miller III: The Heavy Minerals of Florida Beach and Dune Sands. *American Mineralogist* (1945) **30**, pp. 65-75.

[6] J. R. Thoenen and J. D. Warne: Titanium Minerals in Central and Northeastern Florida. U. S. Bur. Mines *R.I. 4515* (1949).

[7] J. L. Gillson: Titanium. *Industrial Minerals and Rocks,* 2nd ed., Ch. 49, pp. 1042-1073. AIME, 1949.

[8] J. L. Gillson: Deposits of Heavy Minerals on the Brazilian Coast. *Mining Engineering* (June 1950) **187**, pp. 685-693.

[9] E. E. Creitz and T. N. McVay: A Study of Opaque Minerals in Trail Ridge, Fla., Dune Sands. *Mining Technology* (1948) **12**, *TP 2426.*

[10] R. V. Spencer: Titanium Minerals in Trail Ridge, Fla. U. S. Bur. Mines *R.I. 4208* (1948).

[11] H. B. Cannon: *Economic Minerals in Beach Sands of the Southeastern United States. Proceedings of the Symposium on Mineral Resources of the Southeastern U. S., 1949.* University of Tennessee Press, Knoxville, Tenn.

[12] L. Alexander and H. P. Klug: Determination of Crystallite Size with the X-Ray Spectrometer. *Journal of Applied Physics* (1950) **21**, pp. 137-142.

[13] B. E. Warren: *Journal of Applied Physics* (1941) **12**, p. 375.

Productivity in Mining Pitching Seams Of the Canadian Rockies

by H. Wilton-Clark

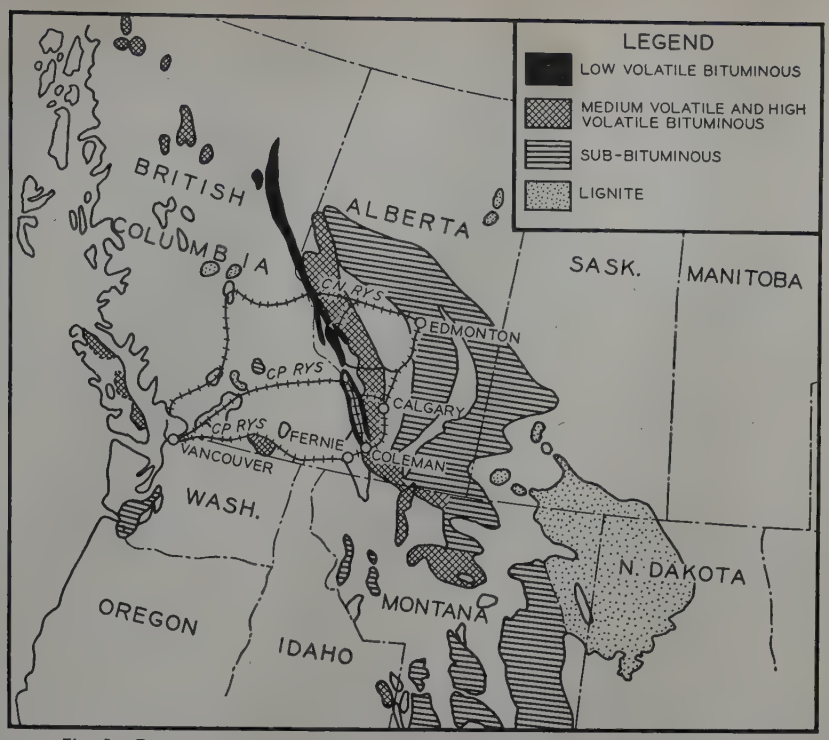


Fig. 1—Extent of coal formations in western Canada and sections of the U.S. Estimates indicate a reserve of 25,000 million tons of bituminous coal.

VARYING in thickness and in number from place to place, coal seams in the Canadian Rockies also range in pitch from nearly horizontal to vertical, sometimes with overturns. Over the entire coal-bearing area there are considerable differences of rank in coals of the same geological age, and there are marked differences in ash content and washability characteristics. Correlation of seams at mining operations within a few miles of each other has often proved impossible. These factors influence mining methods, and, of course, production results.

The oldest coal formations of western Canada are of Lower Cretaceous age, as the carboniferous sediments are marine and contain no coal. Coal is also present in formations of Upper Cretaceous and Tertiary ages. The rank of coal varies from semi-anthracite in some operations in the Kootenay and Luscar formations of the Lower Cretaceous in the Rockies to lignite in the Tertiary fields of the Saskatchewan prairies. Fig. 1 shows the general extent of the formations.

The pitching seams, chiefly Lower Cretaceous, occur in the western belt of the Rocky Mountain foothills and the eastern slopes of the Rockies themselves. (The road to Tent Mt. strip pit, elevation 7000 ft, is shown on p. 832.) Formations extend from the U. S.-Canadian boundary for several hundred miles in Alberta and continue for a similar distance in British Columbia. Present geological estimates show a probable reserve of the order of 25,000 million tons of coal ranging from high volatile bituminous to semi-anthracite, with a large percentage of coking coal, constituting one of the major world reserves of that type. It should be noted that although the quoted estimate probably errs on the conservative side, the question of access to the seams in a mountainous terrain will always be a problem; a wide divergence may exist between actual reserves and the quantities of coal economically recoverable.

An excerpt from the Canadian government *Report of the Royal Commission on Coal 1946* states that conditions favorable to coal formation were intermittent and these intervals relatively brief in comparison with periods in which no coal was formed. Conditions were favorable to the growth or accumulation of vegetation in one area, while fresh water sediments and marine shales were being deposited in other areas. During periods of emergence the coal deposits were subjected to erosion or were covered by coarse sands and gravels from the mountains, whereas during submergence they were covered by fine clays deposited in embayments of the sea. During some of the periods of coal formation, volcanic activity deposited beds of fine volcanic ash and dust with the coal-forming vegetation.

Coal deposits reach their greatest development in the mountains and thin rapidly to the east into the plains area, where they are deeply buried beneath younger sediments. For example, in the Fernie area of southeastern British Columbia there are in places 22 seams having an aggregate thickness of 150 ft in a stratigraphic interval of 3500 ft, whereas at Coleman in the Alberta Crowsnest area the measures are only 800 ft in thickness and contain a maximum of five seams aggregating about 47 ft of coal. At Bellevue, 10 miles further east, the measures are reduced to 430 ft with only three seams aggregating about 37 ft of coal.

Subsequently the tremendous forces involved in the upthrust of the Rocky Mountains produced great displacement of the coal-bearing formations and at the same time a change in geologically young coals from low to high rank.

The floor or footwall of most seams consists of carbonaceous shales, which are almost as adaptable to bending as the coal seams themselves. The seams show very few cleats or cleavage planes and have been so weakened structurally that as mined 50 pct or more will pass through a ¼-in. screen, seldom

H. WILTON-CLARK is General Manager, Coleman Collieries Ltd., Alberta, Canada.

Discussion on this paper, TP 3840F, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, Feb. 16, 1954. Joint Session CIMM and Coal Div., AIME, New York Meeting, February, 1954.

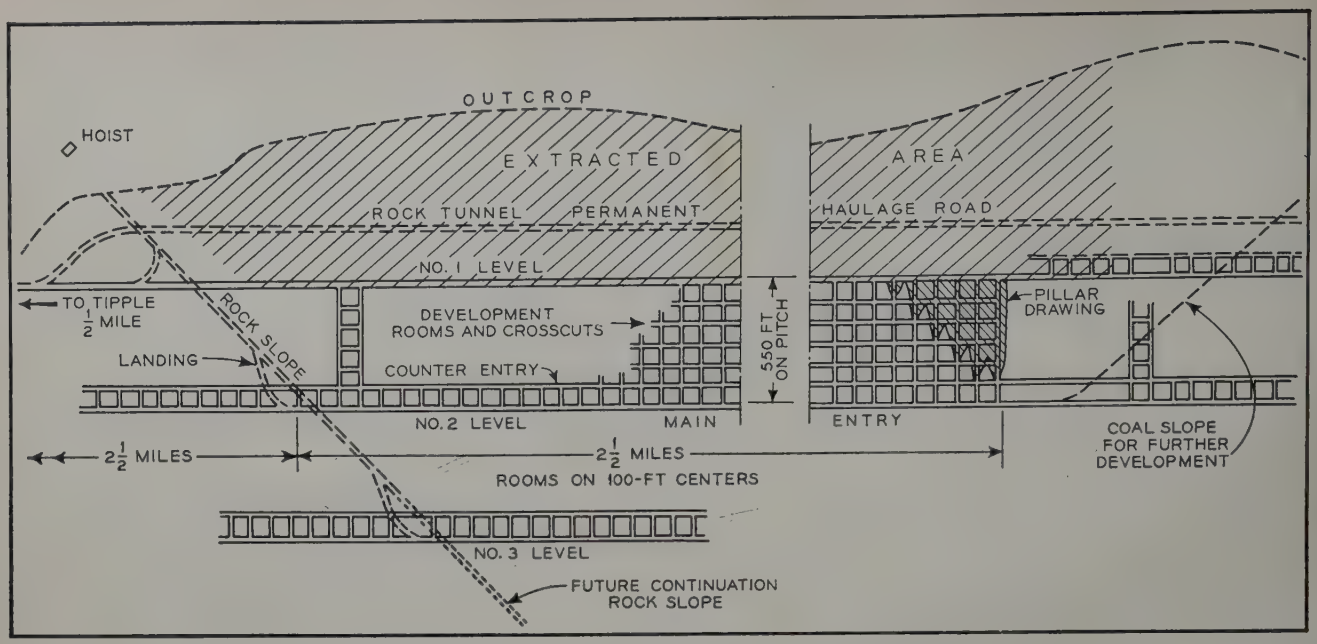


Fig. 2—A typical level in the mine shown on opposite page, Fig. 3.

more than 10 pct being larger than 6 in. Roof is usually shale, at times grading into sandstone and even conglomerate. Shale roof is usually heavy, while sandstone roof, apparently hard and sound, proves to have microscopic fault cracks (as do the shale roofs) and requires systematic timbering. Conglomerate roof is usually good.

Coal mining in the Rockies immediately followed railroad building, the chief market being for railroad fuel. The Canadian Pacific Railway crosses the mountains in two places and the Canadian National Railway in one, with two further branch lines which enter coal-bearing areas. These lines are shown in Fig. 1. The railways generally run east to west, while the coal deposits strike southeast to northwest. Where the pitching seams outcrop adjacent to a railway a mine was opened. As the railways traverse passes resulting from stream erosion the outcrops are usually accessible at valley levels. Most mines, therefore, have been started by drifts in the seams at tipple (railroad) elevation. As the valley sides rise, and the seams with them, the general custom has been to mine coal to the rise of the main entry, until the economics of haulage and travel time to and from the working faces has forced working to the dip. Fig. 2 illustrates the layout of such a mine.

In the last few years, with the advent of diesel locomotives and conversion of steam locomotives from coal to oil, coal markets have steadily declined. The loss in railway market has been taken up somewhat by use of coal in industry and by space heating. Output from the pitching seam mines for the last 14 years is shown in Table I.

The high cost of producing coal and the competition of other fuels have reduced operating mines from 15 to 9 and have forced nearly all mines to supplement their underground production with strip coal from seam outcrops. Strip coal reserves, of course, are very small compared with underground reserves in any given locality.

When coal was mined above the main entry drainage presented no difficulty because of gravity flow to the mine portal. Ventilation was also relatively easy in that airways could be vented to the outcrop when necessary. As the mines have developed dip workings, these conditions have changed. Permanent airways must be maintained for adequate ventilation, usually under conditions of deteriorating roof. The necessity for excellent ventilation is illustrated by the fact that typically the mines give off 3000 to 4000 cu ft or more of methane per ton of coal mined. The strata overlying the coal seams is usually sufficiently fractured to allow considerable water inflow, which varies in quantity in a ratio of 4 or 5 to 1 from June to December. Thus the mine shown in Fig. 2, producing 2000 tons per working day, has a minimum water inflow of 350 and a maximum inflow of 1600 imperial gallons per min, which must be gathered and pumped against 1000-ft head. This mine has two workable seams, the top seam 12 ft thick and the bottom seam varying from 4 to 8 ft. At present nearly all mining is done in the top seam. Above the main entry all coal was removed to the outcrop.

Table I. Output from Pitching Coal Seams of the Canadian Rockies, 1938 to 1952*

Year	Mines, No.	Production from Stripping, Tons	Production from Underground, Tons	Total Output, Tons
1938	15		2,792,896	2,792,896
1939	15		3,186,336	3,186,336
1940	13		3,875,910	3,875,910
1941	13		4,820,536	4,820,536
1942	13		4,981,058	4,981,058
1943	14		4,508,728	4,508,728
1944	14	116,519	4,656,516	4,773,035
1945	13	339,576	3,991,135	4,330,711
1946	13	645,413	4,330,343	4,975,756
1947	12	1,067,043	3,624,441	4,691,484
1948	13	1,611,262	3,359,405	4,970,667
1949	13	1,740,236	3,725,190	5,465,426
1950	14	1,659,898	2,982,556	4,642,454
1951	13	1,553,189	3,120,462	4,673,651
1952	9	1,335,247	3,178,870	4,514,117

* Source: Coal Statistics for Canada for years 1938 to 1952 inclusive.

Below the main entry levels are driven 550 ft apart, measured on the pitch of the seam. Each level consists of a main and counter entry, driven on 100-ft centers. These entries must naturally follow the undulations of the seam laterally, but they are driven

to a fixed grade of 0.6 pct, which allows for adequate drainage and equalizes traction required for empty and loaded trips. If a given district produces much water, a companion entry or water level is driven below the main entry, to avoid drainage ditches in the latter. Where cover is light, for example, less than 1000 ft, rooms with the necessary ventilation crosscuts can be driven as a level progresses and pillars pulled as soon as rooms are finished. As cover increases it has been found advantageous merely to drive the entries to a desirable limit and then drive rooms and pull pillars on the retreat to lessen effects of squeeze.

Fig. 3 shows sections of the entries, with a room and crosscuts. Because the coal is friable, all tight work other than a main entry is driven no more than 10 ft high, as ribs slough, leaving unsupported roof. Entries are systematically timbered with three-piece sets, with high rib and roof lagged. Rooms and pillars are taken out where possible with props and cap pieces, but otherwise with sets and lagging. Roof bolting is now being tried in entries, but so far not in rooms or pillars. It may be mentioned here that timber consumption in the operating mines varies from two to three lineal feet of timber per ton of coal extracted. Entry timbers are typically of 10-in. diam at the top, and props for rooms and pillars have 6 to 8-in. tops. When pillars are pulled the full seam is taken. Fig. 3 also indicates how rooms are driven and pillars are pulled on the retreat. Along one rib of a room a plank chute is built, 3 ft wide and 3 ft high, with the room rib forming one side. The bottom is lined with 22-gage galvanized sheet iron on which coal slides by gravity to the main entry, where it is loaded into mine cars as required. A section along a room is shown in Fig. 3. Mining is by pneumatic picks, with two miners to each room. If the coal is hard it is shot after a mining has been put in with the air picks, and in such case pneumatic rotary coal drills are used. A room 10 ft high and 10 ft wide is usually worked in two 5-ft benches, the upper bench extraction being kept ahead. When rooms and the necessary ventilation crosscuts have been completed, pillars are pulled by skipping from the top crosscut to the top of the room with as wide an area taken out as possible. This is governed by two considerations: 1—condition of roof and tendency to cave, and 2—if the roof happens to be good, by the limit angling of temporary chutes across the pitch, allowing for gravity flow of coal from the face to the point where the temporary chutes feed into the main chute in the room. At the place where the skips diverge from the room a battery of props is built across the room with only sufficient opening for a chute. When the pillar caves the cave will not extend below the battery, and rock falling down the footwall is prevented from going down the room. After such a cave, places are again driven up into the pillar blocks from below the battery, a thin chain of coal being left between the place and the cave. When the top of the pillar is again reached the new pillar split is widened by skipping until again caving occurs. The pillar is thus progressively pulled down to the main entry until only pillar stumps are left; these are usually sacrificed because of excessive squeezing. Under such methods 75 pct of the coal in a level is extracted, rising to 85 pct under good roof conditions. Usually an air hoist is installed in every tenth room, and timber, lagging, brattice, and other supplies pulled up on a sled and distributed by packing crews to adjacent places. Sometimes track and cars are installed, but there is pronounced floor heaving which generally makes the sled more practicable.

As rooms are driven up, ventilation is established by crosscuts. Brattice is carried up a room to ventilate the face until a crosscut is driven, when the lower crosscut is stopped off, usually with brattice and boards. Between the main entry of a level, which may be either intake or return air, and the counter entry acting as the opposite number, double-boarded stoppings are used, with tight slide doors for men to pass through. The chute opening is covered with several thicknesses of brattice cloth,

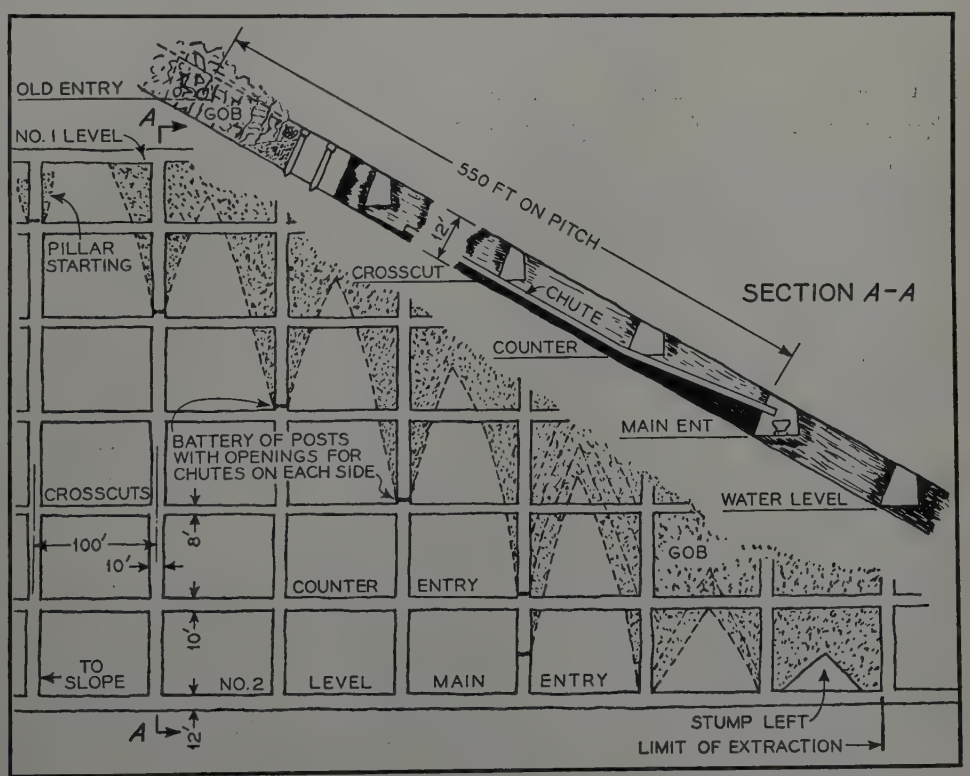


Fig. 3—Sections of entries, with room and crosscuts. Because coal is friable, all tight work other than a main entry is driven no more than 10 ft high. Entries are timbered with three-piece sets and high rib and roof are lagged. Where possible rooms and pillars are taken out with props and cap pieces.

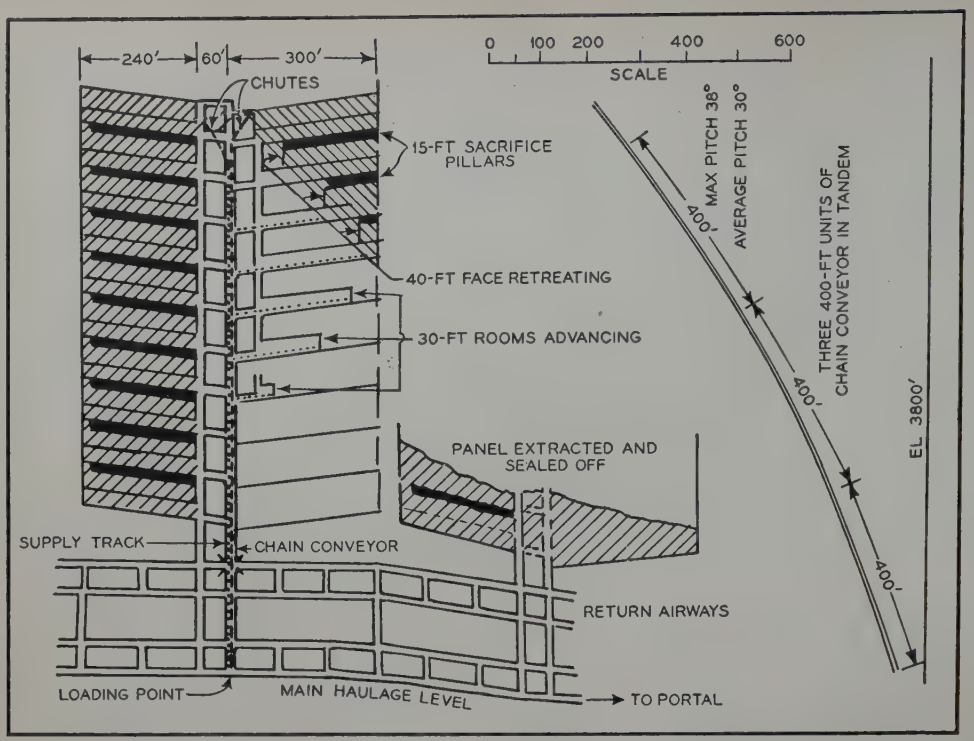


Fig. 4—The panel method for duckbill extraction. Places are driven about 1300 ft, at 60-ft centers, as shown here. Three 400-ft chain conveyors in tandem carry the coal to a loading chute at the main entry.

through which coal will pass. If the place is not abandoned because of pillar removal, this opening is boarded tight on finishing. Levels are driven from main hoisting slopes, such slopes either being driven across the pitch or in rock under the seam. The maximum desirable pitch for such slopes is 22°. At greater inclinations coal will roll off cars if the cars have been loaded level full or more.

As the initial entry is driven from a valley level into mountainous terrain, rapidly increasing cover limits working to the dip. Experience to date has shown that 2500 ft of cover is the present feasible limit. At that point workings are subject to much squeezing, and it is true to say that each entry system at that depth must be literally driven and timbered at least twice, even when a retreat system of mining is used. Also, at 2500 ft seams are subject to violent bumps, making mining at greater depths both uneconomic and extremely hazardous. To develop practical roof control at depths greater than 2500 ft, investigations have been carried on for 5 years by mining engineers, geologists, and physicists, working under the direction of the Dominion government. This is essential, as there are undoubtedly tremendous quantities of coal at great depth in the Rocky Mountains, other than the reserves mentioned earlier. However, it is too early to forecast possibilities of any solution to the depth problem.

Where a seam pitches more than 35° the system described above is still used, with the modification that rooms are angled across the pitch to allow gravity flow of coal. With this method seams can be worked up to the vertical.

Until a few years ago, where a seam was pitching less than 30°, which meant that coal would not flow in chutes, inclines 600 to 1000 ft apart were driven up the pitch. Small air-powered hoists capable of handling one to three mine cars were installed in these inclines and rooms were driven left and right on the strike of the seam. Cars were hauled from incline to face by horses. Sufficient pillar was left between rooms to allow safe mining, and pillars were drawn after rooms were driven. Coal was hand-loaded into cars. Coal from the pillar skips was pushed or bucked down sheet irons to the rooms by manpower. In some mines rooms were driven straight up the pitch as wide as roof conditions would allow, separated by minimum pillars. Rooms were double-tracked with light rail and fitted with a pulley lying on the floor, carrying about three turns of rope and fitted with a brake, so that a loaded car going down the room would pull an empty up. This device was anchored to a prop and moved up as the face progressed.

Under the method described for the mine shown in Fig. 2 production is:

Raw coal per man day overall	4.31 tons
Refuse loss at preparation plant, 11.6 pct	0.50 tons
Net production clean coal per man day	3.81 tons

Costs are divided as shown in Table II.

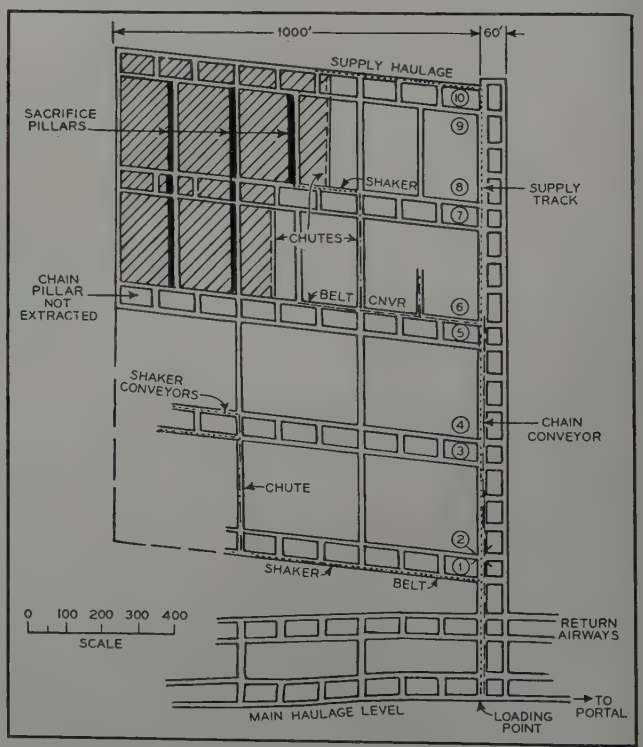


Fig. 5—Modified longwall method on pitch averaging 30°.

Table II. Costs of Mine Production from Pitching Seams by Method Shown in Fig. 2

Category	Total Shifts, Pct
Mining	
Contract miners	29.344
Company miners	1.458
Timbermen	4.666
Timber packers	6.041
Rock dusting	0.401
Ventilation	0.805
Pumpmen	2.101
Pipe fitters	0.338
Handling materials	0.196
Salvage	0.106
Rock handling	0.222
Warehouse and lamphouse	1.107
Maintenance	0.053
Mine timber	0.702
Explosives	0.030
Mantrip	0.007
Tools and nails	0.010
Warehouse	0.288
Administration	0.517
Pit bosses	0.898
Firebosses	3.181
Engineers	
Electrician, timekeeper, consulting engineer, safety supervisor	0.742
Total	53.213

Category	Total Cost, Pct
Contract miners	25.389
Company miners	0.956
Timbermen	3.012
Timber packers	3.796
Special work	0.531
Miscellaneous labor	10.102
Total	43.786

Category	Total Shifts, Pct
Haulage	
Locomotive drivers	5.509
Rope riders	2.585
Tuggermen	0.799
Track layers	2.465
Pipe fitters	0.123
Chute loaders	3.940
Chute repairs	0.149
Hoistmen	1.763
Sandmen	0.186
Car repairs	0.812
Oil and grease	0.437
Warehouse	0.292
Ditching	0.103
Locomotive repairs	0.232
Driver boss	1.090
Stable boss	0.305
Engineers	0.013
Electrician	0.096
Timekeeper	0.172
Roller men	0.302
Special work	3.692
Total	25.063

Category	Total Cost, Pct
Locomotive drivers	3.443
Rope riders	1.613
Hoistmen	1.934
Chute loaders	2.401
Trackmen	1.578
Miscellaneous labor	2.532
Total	13.501

Category	Total Shifts, Pct
Tipple	
Locomotive engineer	0.249
Weighman	0.424
Dumper	0.666
Car coupler	0.610
Crusher tender	0.318
Jig operator, V jig	0.418
Jig operator, B jig	0.553
Air tables	0.060
Greaser	0.311
Cleanup	0.951
Slurry operation	0.828
Furnacemen	0.441
Dryers	0.819
Box car loading	0.371
Scalemen	0.374
Car slabbers	0.474
Slack loading	0.311
Trackmen	0.441
Sampler	0.176
Rejects	0.262
Repairs	0.053
Maintenance	0.656
Special coal loading	0.716
Warehouse, foremen, engineer, electrician, timekeeper, watchman	2.356
Total	12.837

Category	Total Cost, Pct
Preparation plant	5.470
Loading	1.801
Miscellaneous labor	1.489
Total	8.760

Category	Total Shifts, Pct
Additional	
Plant heating and compressors	1.640
Machine shop	3.827
Supervision	
Administration	
Selling	
Office	
Total	3.420
Total labor	100.00

Category	Total Cost, Pct
Plant heating and compressors	1.468
Machine shop	2.229

Summary

Total labor costs*	69.744
Purchase power	7.035
Stores and materials	12.194
Administration, including executive salaries, royalties, taxes, insurance, selling expense, pension costs, welfare fund costs, etc.	11.027
Total	100.00

* Labor costs have been loaded to include Workmen's Compensation Board payments, holidays, Unemployment Commission costs, and medical aid. Costs do not include depletion or depreciation.

The first power tool was the air pick, still widely used, which was introduced in the 1920's together with air drills. Air-powered locomotives were generally used for haulage, as they are today, air being supplied at 700 to 1000 psi from charging stations throughout a mine. Trolleys have not been allowed for safety reasons, but for short hauls or gathering some battery locomotives of permissible type have been introduced. In the last 10 years permissible diesel locomotives have come into increasing use. Vastly superior to air locomotives, diesels have the flexibility of the battery locomotive, if not the power of the trolley. The largest diesels now in use are 15-ton machines with 100-hp engines.

Both in mining and conveying to main entries, development has been slow and arduous. No mobile machinery available as yet is effective on a pitch greater than 5°, which eliminates any self-propelled equipment. It was necessary to attempt mechanization along other lines which would suit conditions in various seams. Examples follow.

Panel Method: As shown in Fig. 4, two places are driven at 60-ft centers, up the pitch from the main entry. In one place a hoist and track is installed for hauling materials, and a chain conveyor is installed along the track, feeding to a loading chute at the main entry. The places are driven about 1300 ft, and three 400-ft conveyors are used in tandem. The top 100 ft of coal is fed to the last conveyor by chute. As the inclines are driven, rooms are broken off on each side. Rooms on the left side are completed and pillars pulled on the advance, and when the inclines

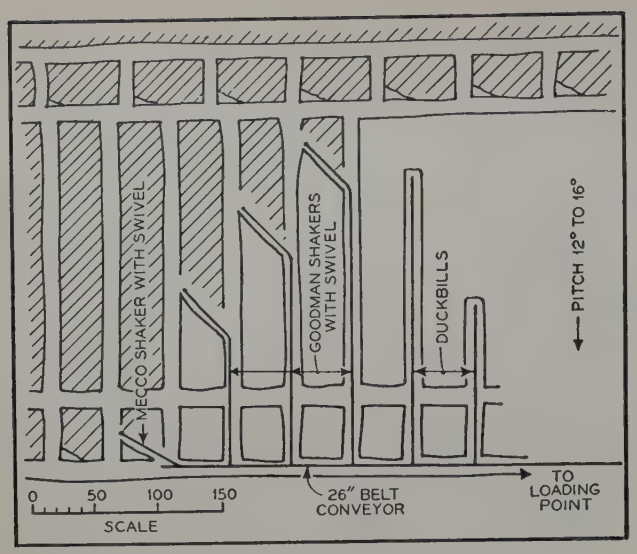


Fig. 6—Seam with poor roof and moderate pitch of 12° to 16°.

are finished, rooms and pillars on the right side are mined out on the retreat. In this instance, where roof is good, rooms are driven 30 ft wide and a 40-ft pillar taken on the retreat, with a 15-ft sacrifice pillar left in. This means that rooms are actually driven on 85-ft centers. Rooms and pillars are cut with shortwall cutters and loaded out to the incline conveyors by duckbills, the coal sliding down the footwall to the duck, which lies along the low side rib of the room. Timbering is by prop and cap piece, on 4-ft centers, which are increased to 4x5-ft spacing immediately over conveyors. In such a system five duckbills are used, four producing and one on the move.

Fig. 5 illustrates this system. One of the two inclines driven up the pitch carries the chain conveyors and hoist and supply track. Rooms are driven off the incline at 60-ft centers, a 250-ft block is left, and another pair of rooms is driven. This system is repeated to the top of the district. Rooms 5, 6, 7, 8, 9, and 10 are driven on an average pitch of 5° for a distance of 300 ft, shaker conveyors being used. A raise is then put through from rooms 5 to 7 and 7 to 10, and a belt installed in room 6, delivering to a chain conveyor in the primary incline. Rooms 5 to 10 are then advanced another 300 ft, with shakers, the coal from room 6 being delivered directly to the belt and the coal from rooms 7 to 10 coming down a chute in the raise to the belt in room 6. The belt is then extended 300 ft, raises are driven through to room 10, and the process is repeated until the full room length is 1000 ft, room 6 having 750 ft of belt conveyor and 250 ft of shaker conveyor. The same method is carried out at the same time with rooms 1 to 4, the coal from room 5 coming out through room 1. At the end of the 1000-ft panel pillar extraction is started in room 8 from the last raise. Coal is cut straight up the pitch with long wall cutters, with an effective cut of 5 ft. The operation commences at midnight when two machine men lower the longwall cutter from the top to the bottom of the face and then proceed to cut up the pitch. They complete the cut and leave the machine anchored at the top of the wall.

On day shift four miners work on the face. A prop bulkhead is erected in the room below the cut coal. This is to allow controlled feeding of the coal through the bulkhead and onto the shaker conveyor in the room. The entire 250-ft face is then drilled and shot. The coal settles down on the footwall but otherwise does not move down the pitch. By removal of bug dust with a scraper at the bottom of the wall, space is made to insert a sheet iron which is spiked to the shale footwall. The coal is then started by barring or by use of air picks. When sufficient coal has been removed, another sheet iron is placed and more coal sent down the wall. This operation is carried on to the top of the wall, the work being finished on the afternoon shift by four more miners whose last job is to remove sheet iron and make the place ready for the cutting crew. Props are set as coal is removed. If from experience a place is judged ready to cave, cutting is commenced in the next raise and pillar extracted toward the cave until only a chain sacrifice pillar is left.

Similarly extraction is carried out in room 6, but following 100 ft behind room 8. If necessary part of the pillar between rooms 7 and 8 is left to hold back gob. Meanwhile timber is taken from the supply incline along the upper room of a wall and then down the pitch to where it is required.

In the lower rooms coal is pulled through room 1, but the pillar between rooms 5 and 6 is left in to stop gob slides from above.

If it is desirable, longwalls can be taken out on the other side of the primary inclines and the area sealed off above the main return airways. The method can be applied only to areas having good roof. It has not been attempted in seams thicker than 8 ft.

Seam with Poor Roof and Moderate Pitch: Fig. 6 shows a method of taking out rooms and pillars in a seam 14 ft thick, pitching 12° to 16°, with friable coal and a weak shale roof having many slips. A pair of rooms is driven at 5° across the pitch and a 250-ft block left to the next pair of rooms. The rooms are driven 300 ft with duckbills and shortwall cutters, and a crosscut is driven every 150 ft. A belt is then installed in the lower room and the room is advanced another 300 ft, and so on, to a total length of 1000 ft. Coal from the upper room is taken to the belt by a shaker conveyor in a crosscut. Rooms are not more than 12 ft wide, crosscuts are 10 ft, and places are driven 10 ft high. Minimum timbering requirements are sets on 4-ft centers, with lagging between collars and also between high side legs to retard sloughing, or, in pillars, props, at 4-ft centers with 3x8x18-in. cap pieces.

From the inby end of the rooms, splits are driven 12 ft wide straight up the pitch with duckbills and shortwalls. When the end split is driven through, the duck and shortwall are taken out, and a swivel installed as indicated in Fig. 6. The pillar is taken out by a series of narrow skips made by air picks and shooting. The pan line is kept alongside the face of the skip and coal either shoveled or shot onto it. The duckbills cannot be used at this stage because of the necessity of props at a maximum of 4-ft centers in every direction. It has not yet been determined whether roof bolting is the answer to utilization of duckbills in these pillar conditions.

The same method is used with split 2. Meanwhile another pan line is installed in split 3, and the duckbill and shortwall from split 1 used in split 3; the duck and cutter from split 2 are used in split 4.

Productivity under any of the methods described above is much the same. Typically, the results are as follows. Costs are divided as shown in Table III.

Raw coal per man day overall	4.60 tons
Refuse loss at preparation plant, 10.5 pct	0.48 tons
Net production clean coal per man day	4.12 tons

Table III. Costs of Mine Production from Pitching Seams by Mechanized Methods

Category	Total Shifts, Pct
Mining	
Contract miners	10.647
Company miners	24.745
Timbermen	3.555
Timber packers	2.036
Rock dusting	0.587
Ventilation	1.484
Pumpmen	0.345
Pipe fitters	0.656
Handling materials	1.225
Washhouse and lamphouse	0.621
Maintenance	0.621
Mine timber	0.311
Warehouse	0.362
Mechanics	0.794
Laborers	1.225
Supplymen	1.363
Administration	0.259
Pit bosses	1.329
Fire bosses	3.986
Engineers	0.569
Electrician	0.173
Timekeeper	0.173
Safety supervisor	0.173
Total	57.239

Category	Total Cost, Pct
Contract miners	8.393
Company miners	17.076
Timbermen	2.349
Timber packers	1.320
Miscellaneous labor	11.498
Total	40.636

Category	Total Shifts, Pct
Haulage	
Locomotive driver	1.881
Rope rider	0.604
Tuggermen	1.087
Track layers	1.363
Pipe fittters	0.863
Chute loaders	0.293
Chute builders	0.414
Hoistmen	2.744
Sandmen	0.362
Car repairs	0.259
Driver boss	0.328
Electrician	0.690
Conveyor runners	3.158
Beltmen	0.880
Conveyor movers	3.814
Laborers	1.363
Pushermen	0.173
Total	20.276

Category	Total Cost, Pct
Locomotive drivers	1.198
Rope riders	0.384
Hoistmen	1.770
Chute loaders	0.189
Track layers	0.873
Miscellaneous labor	8.577
Total	12.991

Category	Total Shifts, Pct
Tipple	
Motormen	0.155
Weighmen	0.794
Dumper	0.293
Car coupler	0.155
Jig operator	0.466
Oiler	0.173
Cleaner	0.155
Dryer	0.310
Box car loader	0.294
Car handler	0.155
Assayer	0.311
Repairs	0.638
Maintenance	1.984
Laborers	2.468
Firemen	0.311
Electrician	1.208
Watchman	0.570
Mechanic	0.690
Foreman	0.673
Truck drivers	0.535
Distributors	0.535
Cat operators	0.345
Total	13.218

Category	Total Cost, Pct
Handling coal	0.994
Cleaning	1.488
Loading	0.282
Miscellaneous labor	5.727
Total	8.491

Category	Total Shifts, Pct
Additional	
Plant heating and compressors	1.243
Machine shop	4.884
Supervision	
Administration	0.552
Selling	0.345
Office	2.243
Total	3.140
Total Labor	100.000

Category	Total Cost, Pct
Plant heating and compressors	0.894
Machine shop	3.184

Summary

Total labor costs*	66.196
Purchase power	5.076
Stores and material	13.904
Administration, including executive salaries, royalties, taxes, insurance, selling and office expense, pension and welfare fund costs, etc.	14.824
Total	100.000

* Labor costs have been loaded to include Workmen's Compensation Board payments, holidays, Unemployment Commission costs, and medical aid. Costs do not include depletion or depreciation.

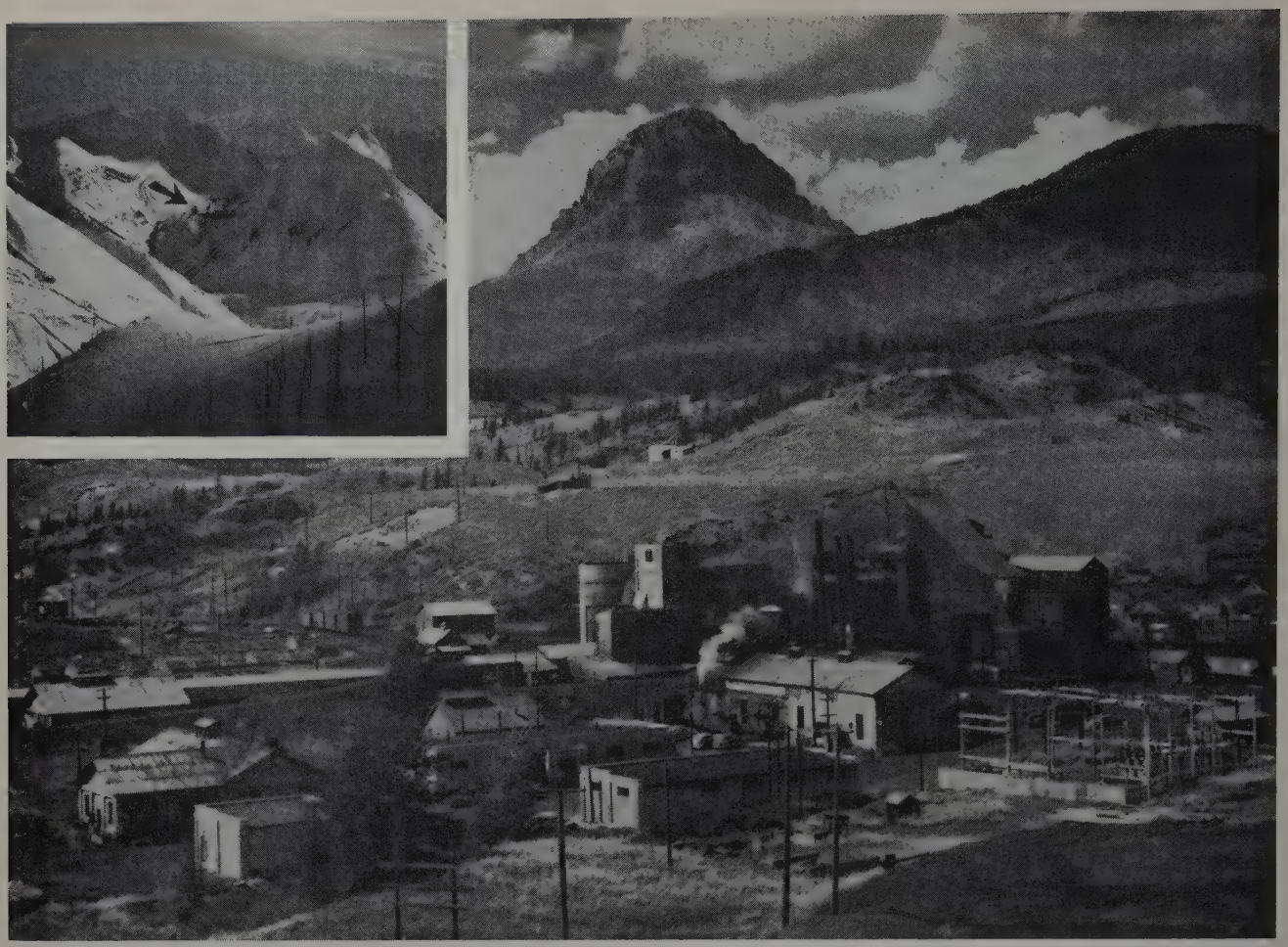
Power costs are less in the mechanized mine than in the non-mechanized mine, which pumps and hoists to a far greater extent.

It is apparent that mechanization has not brought about outstanding results as compared to results obtained with hand-held portable tools, e.g., the pneumatic pick. It should be remembered that gravity is always fighting attempts at mobility. For instance, in a seam pitching 30°, moving a duckbill, pan line, drive, and shortwall cutter from a finished place and setting up in a new location 500 ft up the pitch will take up to 15 manshifts of labor.

However, without conveyor systems, cutters, and loading equipment, mines having seams with a non-running pitch could not compete with mines having seams of running pitch, other factors being equal.

Attempts at mechanization are thwarted for other reasons. The use of face equipment may result in high production per man day when wide places are being worked or pillars taken out, but production drops sharply in tight work places, which may be only 10 or 12 ft wide. Because complete cutting and loading equipment must be installed for each place, cost is high per ton produced. Equipment is utilized for only part of each shift because of timbering requirements. For example, under heavy roof a set must be put up as soon as coal has been removed to allow for it and the roof lagged from set to set.

Also, during the life of the industry a wage structure has been developed which includes contract rates for miners. In a mechanized mine the face worker is on day wage. So far no incentive system has been developed which is satisfactory. Equipment is not mobile and requires much manual labor to operate and move. At the same time the investment is very high per ton of coal produced. If the operator's investment in machines is to be justified, it appears necessary to establish an incentive rate that will encourage the worker to obtain maximum production from machines.



The surface plant at International mine, Coleman Collieries Ltd. at 4200-ft elevation. Inset shows the road to Tent Mt. strip pit (indicated by arrow) at 7000-ft elevation.

It is regrettable that mechanization does not show up to better advantage. The question arises as to what can be done to improve the situation. Great effort has been made in attempting to adapt equipment to pitch conditions, but available equipment is not the answer. Chiefly, lightness is required, and conveyors, whether belt structures, shakers, or flight conveyors, including the drives, must be developed to have capacity and strength with much less weight. As mobile equipment is out of the question, semi-mobile cutters similar to the German *Eikhoff* appear desirable. This small light machine includes self-propelled crawler treads and an inbuilt winch to pull itself to the face, with hydraulic jacks for holding position and a retracting universal cutter head.

Even with light equipment, tight work will be a problem. A place which can be held only 10 or 12 ft wide may be cut in a few minutes, yet 2 hr may be required to maneuver a shortwall cutter to the face and away again. Experiments are now being carried out in shooting complete faces from the solid by suitable patterns of split-second delay action shots. For tight work there is a promising place for such methods, provided that there is adequate ventilation, proper rock dusting or wetting, and correct shooting equipment and materials, handled by competent shotlighters.

As in all mines, transportation is steadily improving. Light-weight high-capacity mine cars with roller-bearing wheels are hauled by diesel locomotives, traveling on better track.

Reference to cost distribution shows that a considerable percentage of total manpower is utilized on surface. To date this has been unavoidable, as the coal must be washed, and in these climatic conditions dried, a major problem because the generally friable coal runs largely to fines. Preparation plants are adequate but because they have been developed over many years by additions and changes large staffs are necessary. Future mines with well-designed plants will undoubtedly improve greatly on throughput per manshift.

Electric power underground replaces compressed air at a saving of 5 to 1 and greatly improves machine operation. For percussion machines such as picks, drills, and rock drifters, compressed air is better than electricity, but to keep piping at a minimum it is probable that in concentrated production areas continued use of electric-powered portable compressors will be preferred.

Experimental drilling of crosscuts and breakthroughs has been conducted with large-diameter augers. Future use of such equipment is problematical because of the relatively cumbersome machinery and the requirement that a seam be uniform and free of *sulphur balls*.

Although it is not foreseeable that overall costs or output per man day can approach production under flat seam conditions, it is possible that output can be increased 50 pct, i.e., from 4 to 6 tons, by use of adequately designed lighter equipment and development of semi-mobile machines.

Acknowledgment

Acknowledgment is made to T. G. Ewart and T. H. Wilson of Crow's Nest Pass Coal Co., Fernie, B. C., and to A. E. Graham, E. Price, and R. Upton of Coleman Collieries Ltd., Coleman, Canada.

Collection of Laboratory Dusts

by Benny Langston and Frank M. Stephens, Jr.

Although little information is available concerning small-scale equipment for dust collection in laboratories, it is possible to adapt standard equipment for laboratory use. Dust from laboratory processes may be collected by cyclone separators, filters, electrostatic separators, scrubbers, and settling chambers.

IN recent years much attention has been given to recovery, treatment, and disposal of dusts discharged into the atmosphere from operations of industry. Considerable data has been accumulated on both operation[1-4] and design[5, 6] of dust-collector equipment for commercial installations. On the other hand, there is almost no published data on design and construction of small-scale equipment to handle dust problems that arise in the ore-dressing laboratory.

Dust-collection equipment such as multiclones, single-cyclones, scrubbers, chemical and mechanical filters, settling chambers, and electrostatic separators has proved its efficiency for collecting dust in industry. In the laboratory, however, the engineer is faced with the problem of collecting small quantities of dust, inexpensively, without diverting the major effort from the metallurgical problem to the problem of collecting dust produced by the process.

For most applications standard dust-collection equipment is too large for use in the laboratory; however, for control of dust in large working areas it is often possible to use a standard dust collector, such as an air filter, with branch ducts to eliminate a health hazard. For example, the well-furnished sample-preparation room containing small jaw crushers, rolls, and pulverizers, in addition to the riffles and screens necessary for preparation of samples, presents a perennial source of dust.

The authors' experience has shown that a combination system consisting of overhead branch ducts to the individual equipment and floor ducts with grills, where applicable, connected to a central dust collector effectively removes dust generated in preparation of samples.

Fig. 1 is a sketch of a downdraft dust-collector for table installation. Similar systems can be built with floor grids. For portable equipment such as laboratory vibrating screens this type of installation with a steel grill to support the heavy load is reasonably efficient. Overhead branch ducts to individual crushing and grinding equipment, although efficient, must be carefully controlled by dampers to prevent excess loss or a change in the composition of the sample. Change in sample composition can result from excess velocity, which causes selective removal of constituents of low specific gravity.

Fig. 2[7] shows the theoretical effect of terminal velocity on spherical particles of different specific gravities in air and water under action of gravity. Fig. 3 shows the effect of air velocity on composition of $CaCO_3$ coal mixtures.

Although the entrainment of dust particles in a moving air stream is the basic mechanism by which all dust-collection equipment functions, usually intake velocity of the dust-collection system must be controlled to prevent loss of part of the sample.

As an example of what may happen when excess velocities are used, a mixture of 50 pct coal and 50 pct limestone was crushed to —10 mesh and fed to a pulverizer equipped with an overhead dust-collection system. Fig. 4 shows the overhead dust-collection equipment used in this test. The pulverizer was set to give a product 95 pct —100 mesh in two stages. Velocity of air passing over the lip of the pulverizer was measured with an anemometer. After grinding, the finished product was analyzed to show the amount of calcium carbonate present. Fig. 3 shows graphically the increase in calcium carbonate as velocity through the dust-collection duct was increased. These data show that at a velocity of 1 ft per sec little if any of the coal was entrained by the overhead draft. At the maximum velocity, about 6.5 ft per sec, approximately 7 pct more coal was entrained than calcium carbonate.

From an operating standpoint, this problem can be remedied easily by dampering the line to control velocity. The lowest velocity commensurate with satisfactory dust control should be used to prevent excess loss and, in some cases, selective dust loss.

Collection of Dust in Laboratory Processes

As in industry, the engineer desires to collect efficiently the dust produced by processes being investigated on a laboratory scale. However, in the collection of laboratory dusts he is faced with two additional problems: 1—The volumes of gas and the quantity of dust that must be recovered are small when compared with the capacity of standard dust-collector equipment, which must be scaled down in design except for collection of dust from large pilot-plant operations. 2—In addition, because of the variety of problems studied in the process laboratory, the engineer cannot design today a dust collector that will meet the conditions imposed by the processes of tomorrow. Sometimes, therefore, he must compromise collection efficiency to minimize the cost of fabrication and the amount of time diverted from the metallurgical to the dust-control problem.

For collection of dust from laboratory processes a cyclone separator, filters, electrostatic separators, scrubbers, and settling chambers can usually be adapted for small-scale operations. The following

B. LANGSTON, Junior Member, AIME, and F. M. STEPHENS, Member AIME, are associated with Battelle Memorial Institute, Columbus, Ohio.

Discussion on this paper, TP 3805B, may be sent, (2 copies) to AIME before Oct. 31, 1954. Manuscript, Feb. 23, 1954. New York Meeting, February 1954.

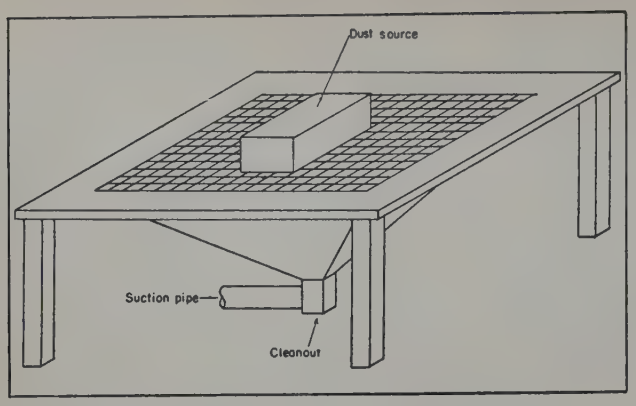


Fig. 1—The downdraft method of dust control.

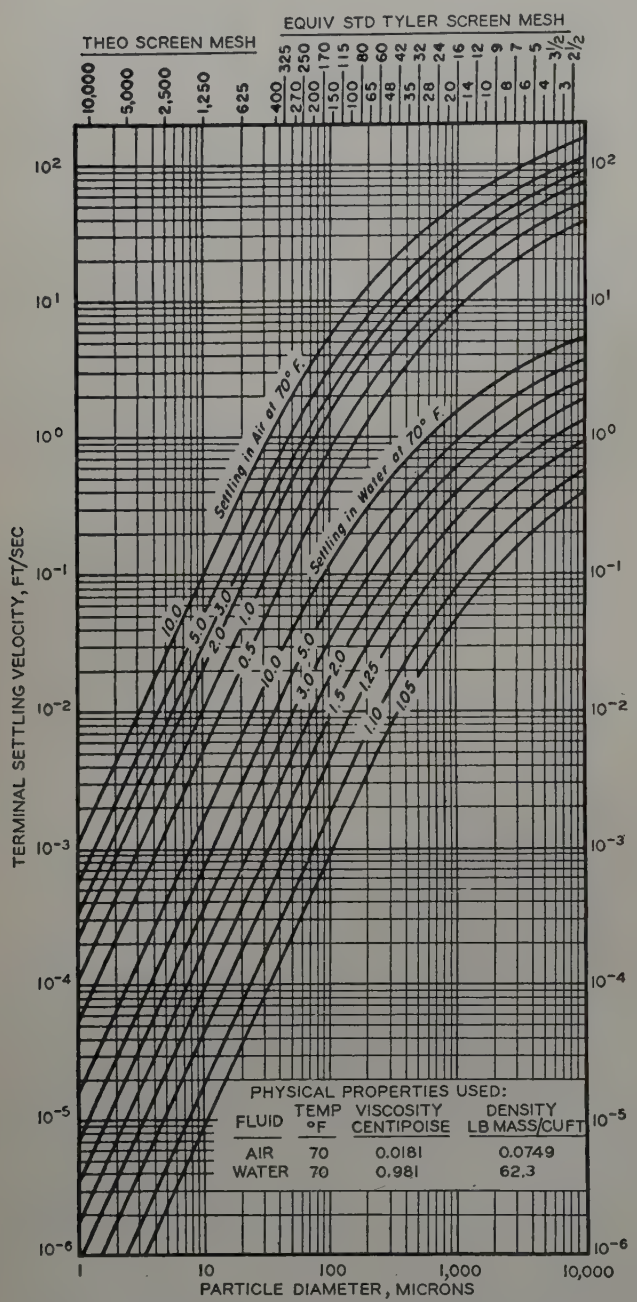


Fig. 2—Terminal velocities of spherical particles of different density settling in air and water at 70°F under the action of gravity. Numbers on curves represent true (not bulk or apparent) specific gravity of particles referred to water at 4°C. The Stokes-Cunningham correction factor is included for fine particles settling in air.

sections describe some of the fundamental concepts involved in the design and fabrication of small units of each of these collectors. Several examples are given to show problems characteristic of a small-scale dust collector.

Cyclones: The size of a cyclone is governed primarily by volume of gas and sizes of particles in the dust load, but the main variable determining the collecting efficiency is the centrifugal force applied to the gas stream. In the laboratory where small volumes of gas are encountered and reasonable collection efficiencies are desired, the cyclone must be small in diameter to obtain the pressure drop and applied centrifugal force required for efficient collection of dust.

The fabrication of laboratory cyclones is simple in that black iron sheet, which can be sheared and rolled easily, will withstand the temperature and corrosion conditions normally encountered in laboratory dust collection. Fig. 5 shows the proportions generally employed in construction of a cyclone, as well as a modified design of the standard type. Experience has shown that some improvement can be achieved in the collection efficiency obtainable in a small cyclone by starting the tapered section one diameter from the top of the cyclone rather than the normal two diameters.

The small cyclone does present certain operating problems. For example, because of the small opening for discharging the dust, fine dust with a high angle of repose tends to bridge and plugs the discharge. Laboratory expedients for preventing bridging include installation of external vibrators or internal moving chains sealed by a packing gland. These can be operated periodically to keep the dust moving. Another problem encountered in operating small cyclones is the plugging of the inlet tubes, and it is often advisable to equip the cyclone with access tees to allow for periodic cleaning of the entrance line.

Filters: Cloth bag filters have been used for many years for the collection of dust. Once a suitable filtration bed has been established by deposition of dust on the cloth surface, collecting efficiency is reasonably constant under variable operating conditions. As in industrial applications, the chief concern in the application of bag houses to collection of laboratory dusts is the estimation of resistance of the filter, the time cycle for cleaning, and the selection of proper filtering media.

In the construction of a laboratory bag filter, the most important factor is selection of the proper filtering media. Usually this automatically determines the filter resistance and the time cycle for cleaning. The writers' experience has been that temperature of the gases, particle size of the dust, corrosive properties of the gas and dust, and humidity are the main factors to be considered in selection of the filter media.

When a bag house is used for laboratory dust collection, the dust-laden gases pass into socks constructed from material which will stand the conditions of temperature and corrosion. The exhaust fan sucks the gases through the filter media and dust is collected in the hopper. An advantage of this method is that proper selection of the exhaust system, uneconomical in a commercial plant, increases flexibility of the equipment for use in the laboratory. Another advantage of the system is that passing the gases downward collects the dust deposited on the

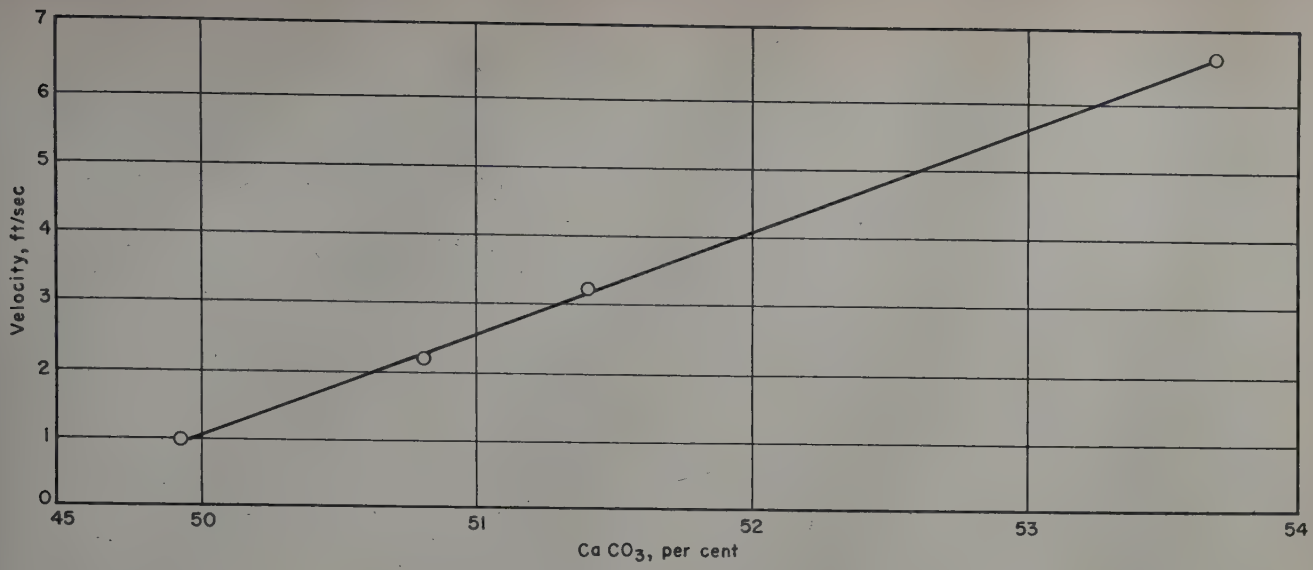


Fig. 3—Effect of air velocity on composition of $CaCO_3$ coal mixtures.

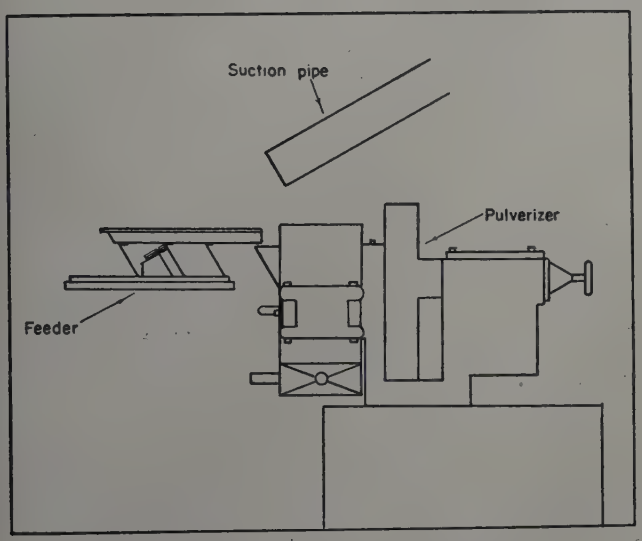


Fig. 4—Equipment for overhead dust collection.

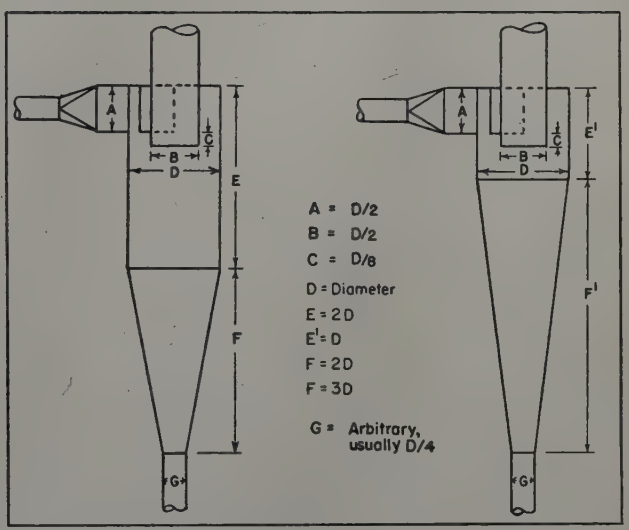


Fig. 5—Standard collector (left); modified design (right).

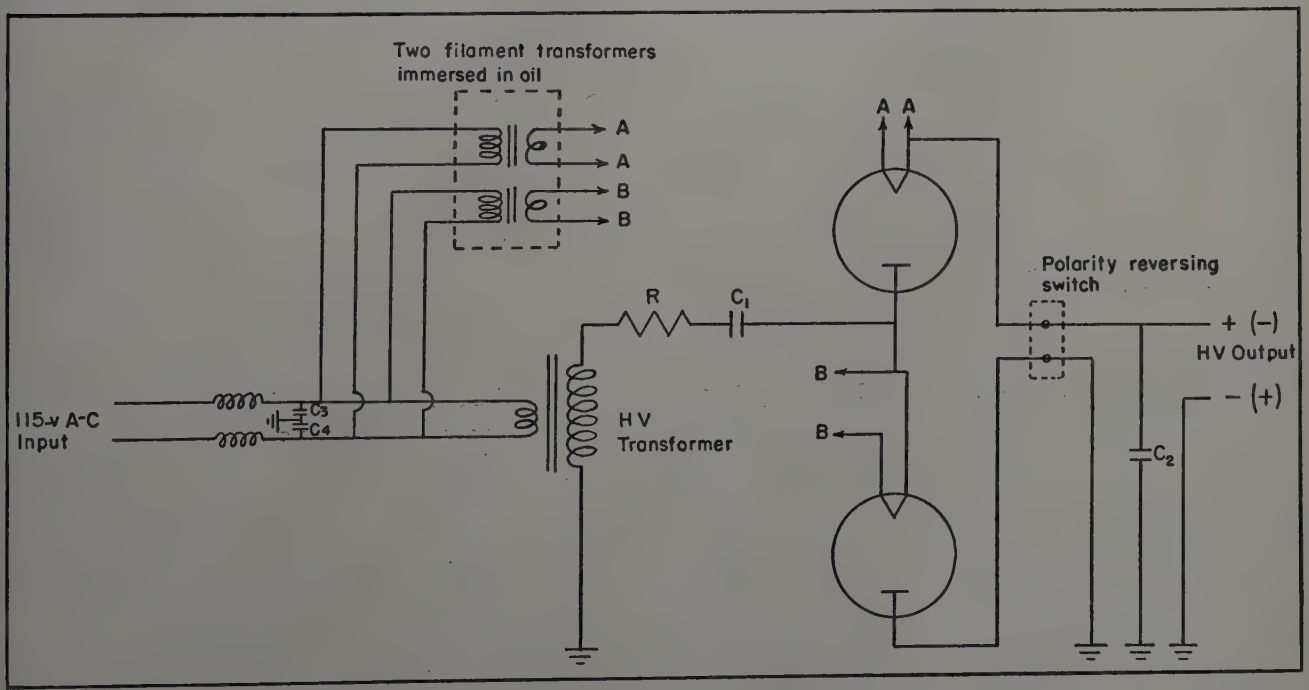


Fig. 6—Circuit diagram for high-voltage power supply.

filter media without passing it through an upward current of gas.

Recently the development of woven asbestos and glass has materially improved construction of the laboratory bag house for the collection of dust from gases having a temperature as high as 275°F and requiring an acid-resistant filter media.

Although mechanical tappers can be used to clean the bags, in the laboratory where continuous cleaning of the bags is a luxury, manual tapping decreases the cost for construction of the bag house. In some processes where gases are cool and noncorrosive an ordinary vacuum cleaner can be employed as a bag filter to recover small quantities of dust.

Electrical Precipitators: For general performance under widely varying conditions the electrical precipitator has no equal. Admittedly the dust load should not exceed 10 grains per cu ft of gas for maximum collection efficiency. However, if dust concentrations greater than this limit are encountered, installation of a cyclone in front of the Cottrell will reduce the dust load to a point where collection efficiencies approaching 99 pct of the dust in the gas stream may be obtained.

The electrical precipitator consists of four principal parts: 1—a source of high voltage, 2—the high-voltage ionizing electrodes and associated collecting electrodes, 3—equipment for collecting the dust, and 4—a shell to house the precipitator. One of the major factors in the construction of a laboratory precipitator is the power source. In a tube-and-wire precipitator the shell and the discharge hopper to collect the dust can be constructed readily from rolled black-iron sheets. It may be either a single tube or twin tubes 6 in. or less in diam, depending on the volume of gas that must be treated. The ionizing electrode usually consists of a Nichrome or Chromel A wire of 22 gage or smaller. Normally wire-and-plate precipitators are not used in the laboratory unless relatively large volumes of gas must be treated.

Although the advice of an engineer familiar with electronics is advantageous when a power source is constructed for a laboratory precipitator, it should be possible for the average engineer to build a suitable power source by following the circuit diagram shown in Fig. 6. The diagram shows a unit that can be constructed from materials available to the average laboratory.

An 8000-v transformer, originally designed for igniting an oil burner, is used as a source of high-voltage power. Two 30,000-v, 500-micromicrofarad television filter condensers (C_1 and C_2) are used to isolate the transformer secondary from the load in the circuit, thereby preventing an overload when the high-voltage output is shorted, as so frequently occurs under operating conditions. Secondaries of the filament transformers are insulated from primary windings and ground to withstand the high-voltage output. The two transformers used for this purpose, both Merit Coil & Transformer Corp. No. P2942, are immersed in oil for additional protection. To protect the circuit when the Cottrell is discharged, two 0.05-microfarad condensers and two choke coils, consisting of 100 turns of No. 24 cotton-covered enameled wire on a ½-in. core, were placed in the power-input line. The rectifier tubes are Type 8020. This circuit will provide a 14,000-v power supply for use with single or twin-tube precipitators, varying from 4 to 6 in. diam.

Care must be taken in the installation of the ionizing wire to prevent shorting of the circuit by build-up of the dust between wires and shell. This can be remedied by placing a small vibrator on the precipitator shell, but the authors' experience has been, particularly with up-draft precipitators, that continuous vibration lowered collection efficiency. For this reason the vibrator is operated only at periodic intervals, depending on the dust load, for sufficient time to relieve dust flocculated on the shell.

Often it is advantageous to have cleanout ports near the entrance and exit ports of the Cottrell. For example, in the collection of dust from a sulphating roast, gases contain iron oxide as dust and sulphur trioxide as a mist. Under certain temperature conditions the iron oxide and sulphur trioxide form ferrous sulphate, which plugs the Cottrell and stops all passage of gas until the unit is cleaned out.

Scrubbers: Although in many cases the scrubber is efficient as a dust collector, several disadvantages limit its use in the collection of laboratory dusts. Large volumes of water or chemical reagent must be passed through the scrubber to keep the solids from plugging the discharge port. In the laboratory, where it is often desirable to recover and analyze the dust, use of a scrubber requires filtration of the solution to recover the solids. In the case of chemical reagents, any corrosive solution must be neutralized prior to disposal down laboratory drains. Also, if the dust contains soluble values and the process requires analysis of the chemical solutions to obtain metallurgical balances, the large volumes of solution give such a low concentration of values that accurate determinations are difficult if not impossible. In essence, although the scrubber has proved a good commercial dust collector, in the laboratory other methods appear to have the advantage.

Settling Chambers: The settling chamber basically reduces the velocity of the gas stream from a velocity which entrains dust to one at which the solids will settle. For this reason it is probably the simplest of all dust collectors. However, because its practical applicability is limited to removal of particles larger than 50 to 100 microns, its use in the laboratory is limited to applications where it is not necessary to remove all the dust prior to exhausting the gas.

Conclusions

As indicated by the discussion on each of the individual types of dust-collecting equipment, there is no single solution to the problem of collecting the small quantities of dust produced by laboratory processes. However, by utilization of available equipment and application of ingenuity and good engineering judgment it is usually possible to overcome the problems that exist in collection of laboratory dusts.

References

[1] W. H. Dennis: Dusting in the Copper Smelters. *Mining Magazine* (1947) **77**, pp. 16-22, 78-80.

[2] W. H. Dennis: Fume and Dust in Lead Smelting. *Mine and Quarry Engineering* (1947) **13**, pp. 237-242.

[3] L. C. McCabe: Control of Open Hearth Fumes. *Industrial Engineering Chemistry* (March 1951) 89-A to 92-A.

[4] H. V. Welch: The Fume and Dust Problem in Industry. *Journal of Metals* (1949) **12**, pp. 213-236.

[5] C. F. Montross: Entrainment Separation. *Chemical Engineering* (1953) **10**, pp. 213-236.

[6] J. M. Dalla Valle: Dust Collector Costs. *Chemical Engineering* (1953) **11**, pp. 177-183.

[7] J. H. Perry: *Chemical Engineers Handbook*, 3rd ed., p. 1017. McGraw-Hill Book Co., New York, 1950.

LITHIUM

Northeast Brazil Is Potential Source

Of the 61 dikes examined some yielded cassiterite, tantalite, and beryl; eight contained spodumene; and six carried amblygonite. Two mines stockpiled spodumene as a byproduct. Only zoned pegmatite dikes carried lithium minerals.

by W. B. Mather

GEOLOGICAL studies have revealed the occurrence of hundreds of pegmatite dikes in northeast Brazil, Fig. 1, in the three states of Rio Grande do Norte, Paraiba, and Ceara. Heretofore data have not been available on the possible production of lithium minerals from these areas, and in March, April, and May 1953 the author undertook a reconnaissance survey covering a cross-section of the pegmatites. Sixty-one pegmatites were examined.

The pegmatites of Rio Grande do Norte and Paraiba are located in an arid region (Seratao), and except in the most southerly portion there is a minimum of vegetation and overburden. The region is easily prospected and relatively accessible by jeep or truck. The pegmatites examined in Ceara are in an area of lush vegetation along the flanks of the Jaqueribe River valley. It is therefore more difficult to prospect, and the dirt roads, as a means of access, leave much to be desired. For all practical purposes rail transportation, except in Ceara, is not available.

The rocks of the area[1-4] are composed of Pre-Cambrian schist, gneiss, quartzite, and marbles. They have been intruded by granites, monzonites, and diorites and cut by quartz veins and pegmatites.

Pre-Cretaceous erosion reduced the area to a peneplain upon which Cretaceous or younger sandstones were deposited. Recent erosion has left only a few remnants of these younger rocks. In most of the Rio Grande do Norte-Paraiba area the pegmatites were more resistant to erosion than the enclosing schists and therefore stand out in relief. This is not true in the rolling country of Ceara, where the pegmatites lack topographic expression.

With regard to structure and lithology, the pegmatites of northeast Brazil are divided into two types, uniform and zoned. Of the 61 examined, 19 belong to the uniform and 42 to the zoned type. Uniform pegmatites are normally long, narrow, and tabular, with sharp contacts. They are composed of an intimate mixture of feldspar (microcline or orthoclase), mica (biotite, phlogopite, or muscovite), and quartz. The grain size[5] varies from fine at the contact to coarser in the interior. They are not considered a commercial source of pegmatite minerals, although efforts have been made to produce green beryl of gem variety. This type of dike is often abundant in schists and gneisses close to granite intrusives.

The productive dikes — lenticular, pod-shaped, or irregular in outline, with sharp contacts — are of the zoned variety. Zone contacts are gradational, except for those with the quartz core. Zones[6] are identified as follows, see also Fig. 3.

Border Zone: This outer zone, in contact with the country rock, is composed chiefly of fine-grained muscovite and biotite, with lesser quantities of fine-grained microline and quartz. It is the narrowest zone, usually measurable in inches.

Wall Zone: Lying between the border and intermediate zones, the wall zone is composed of medium-grained feldspar (often microcline), muscovite, and quartz. Usually it is the widest zone. Widths of 50 ft were noted.

Intermediate Zone: Lying between the wall zone and core, the intermediate zone is composed principally of coarse-grained microcline (occasionally with some orthoclase and albite). It may also carry minor amounts of coarse-grained quartz and fine to medium-grained mica. The width varies from less than 3 ft to more than 30 ft.

Core: The center of the dike is composed almost entirely of massive quartz, occasionally with minor amounts of fine-grained muscovite. The width reaches 30 ft.

Lithium and other minerals of potential economic value are found in the following zones:

Spodumene	Zone 3 and outer part of zone 4
Amblygonite	Zone 3 and inner part of zone 2
Cassiterite	Zones 2 and 1
Tantalite-columbite	Zones 2 and 3
Beryl	Zone 3 and outer part of zone 4. Occasionally in zone 2
Mica	Zones 2 and 1

Weathering of spodumene and amblygonite produced sharply contrasting results. Spodumene has weathered more rapidly and to greater depths than the other pegmatite minerals. Amblygonite is one of the minerals most resistant to weathering.

W. B. MATHER, Member AIME, is Chairman, Mineral Technology Dept., Southwest Research Institute, San Antonio, Texas.

Discussion on this paper, TP 3869H, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript, Feb. 18, 1954. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 836. The intervening non-Transactions pages appeared in Mining Engineering.

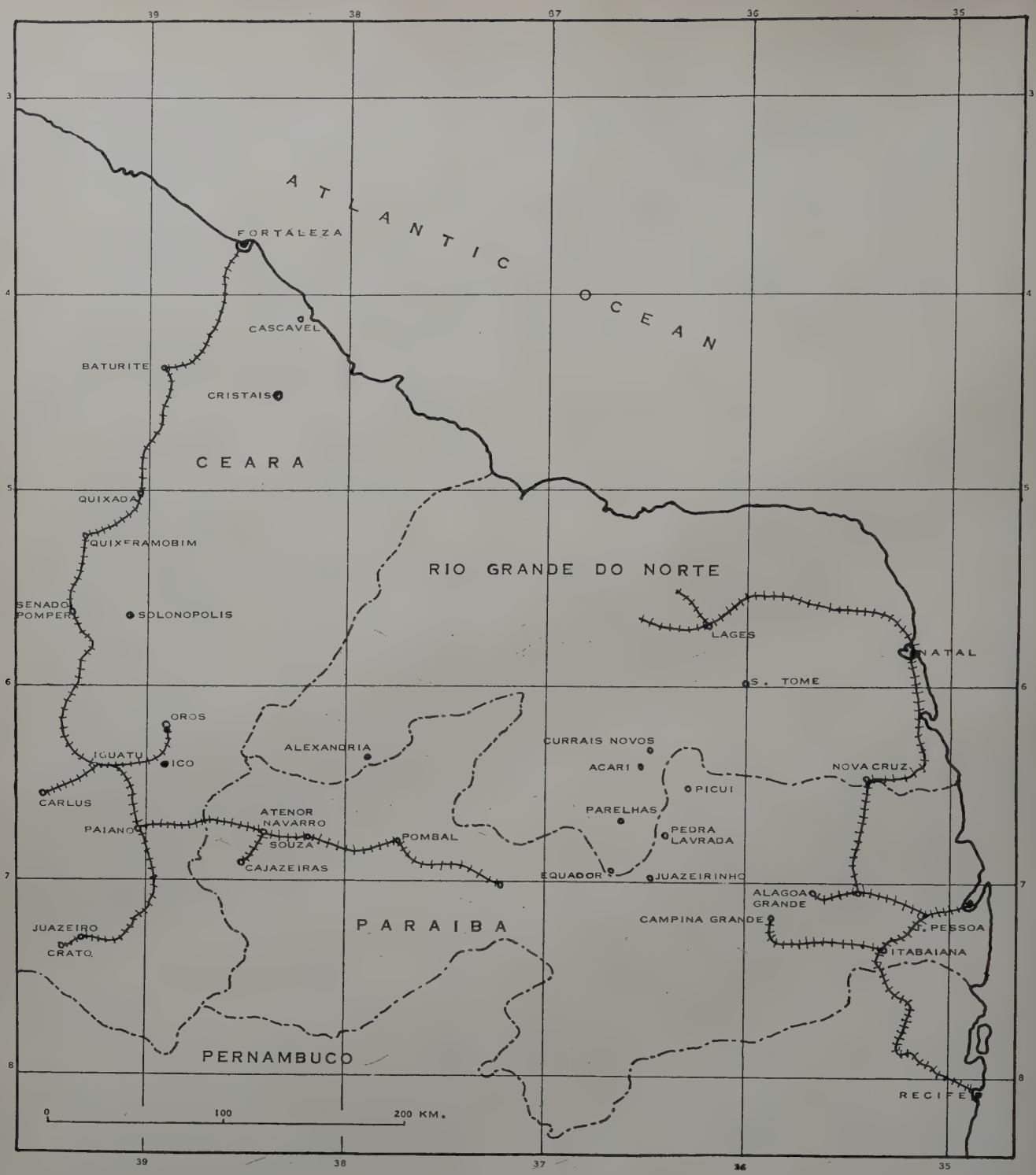


Fig. 1—Index map of northeastern Brazil.

The following minerals were also identified, usually in zone 2: pyrite, lepidolite, tourmaline (also zones 1 and 3), apatite, chalcopyrite, chalcocite, malachite, carnotite, ilmenite, magnetite and arrojadite (also zone 3). Additional minerals identified[7] in these pegmatites are: microlite, simpsonite, rare earth tantalates and niobates, monazite, pitchblende, garnet, metallic bismuth, zircon, lazulite, rhodonite, and molybdenite.

The principal lithium-bearing pegmatites were found in two general areas, Fig. 1. The first extends across the state line from Parelhas and Acari in Rio Grande do Norte on the west to Pedra Lavrada and Juazeirinho in Paraiba on the east. In this report these have been further subdivided geographically into deposits in Rio Grande do Norte and in Paraiba. The second is west and north of Solonopolis in the Cachoeira-Quixeramobim area of Ceara.

The third area, the Cascavel-Cristais district of North Ceara,[8] is known to contain lithium-bearing pegmatites (amblygonite). These were not examined because they have not been mined for several years and workings were reported caved and flooded.

Of the seven pegmatites examined in Paraiba, see Fig. 2, four contained lithium minerals. Two of these, Seridozinho and Pedras Pretas, are located in an

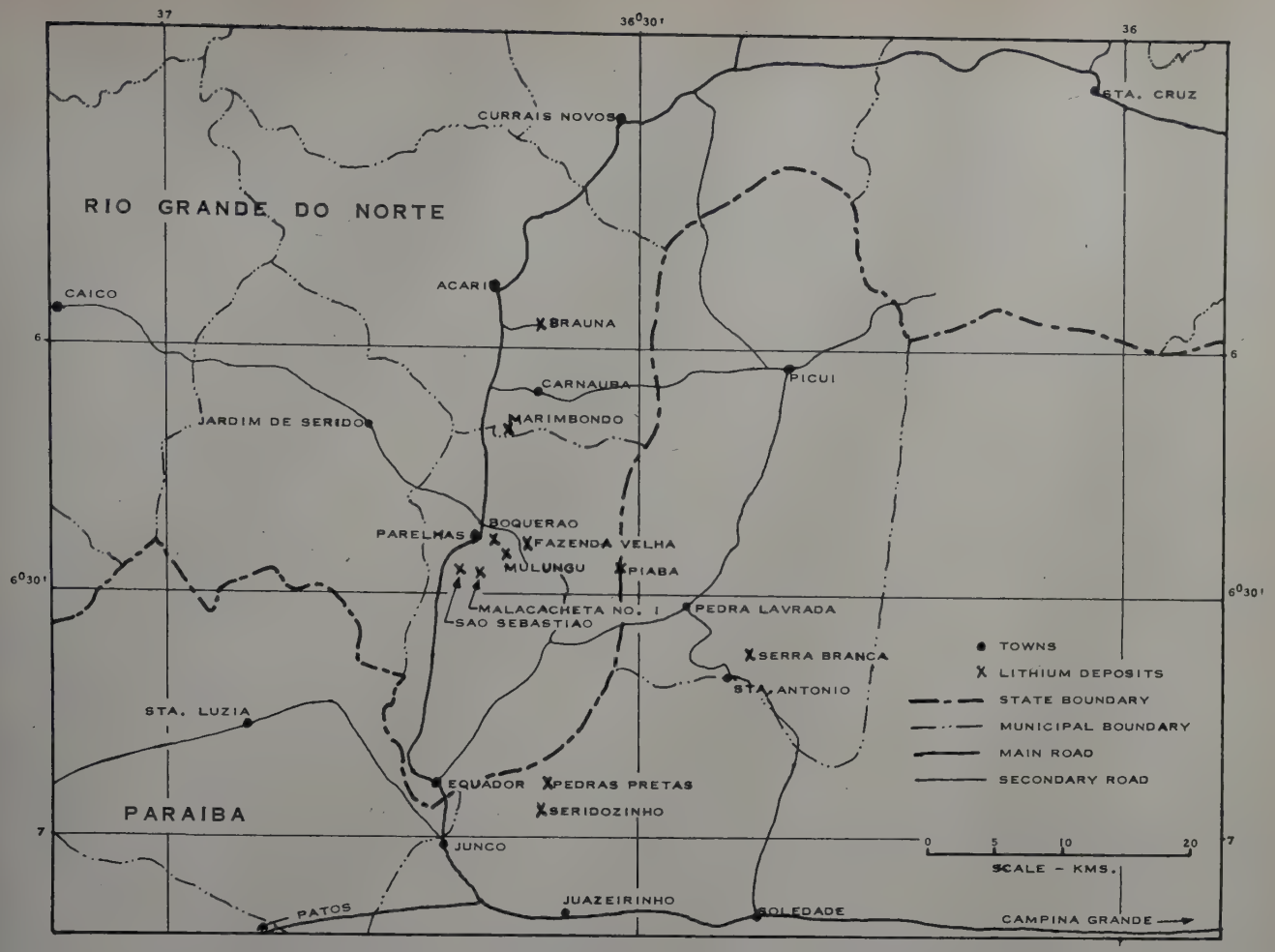


Fig. 2—Index map of lithium deposits in Rio Grande do Norte and Paraiba.

area covered by a mantle of topsoil. The other two are located in a rocky terrain practically devoid of vegetation and overburden.

Seridozinho: Fig. 4 is a sketch of the Seridozinho workings. The mine is located in the southern part of the area on the Borborema plateau, 6½ miles northwest of Juazerinho. One ton of hand-sorted tantalite-columbite and one ton of cassiterite were produced per month. Current beryl production figures were not available. Mining of spodumene was incidental to production of the other minerals. It is reported that total spodumene production to date has been 800 to 1000 tons. Several stockpiles, see Fig. 5, containing an estimated 300 to 400 tons were examined.

During World War II two open pits were brought into operation as a result of tantalite-columbite mining. The south pit, 600 ft long, averages 75 ft wide and 30 ft deep. The north pit is 250 ft long, 90 ft wide, and 80 ft deep. A test-pitted area, 125 ft long and 105 ft wide, averaging 15 ft deep, lies between the two pits. The exposed length of the dike, therefore, is nearly 1000 ft, the width varying from 60 to 105 ft. Present mining operations consist of hand-sinking gopher holes in the floor of the main pit. The maximum depth of the original open pits was 30 ft. The narrow gopher holes extended this to over 45 ft. The owner stated that the old test pits, now caved, indicated a northeast extension of the dike for an additional 500 ft.

The pegmatite has intruded a highly contorted mica schist. The general strike varies from N 40° E to N 60° E. The dip of the upper part of the east wall varies from 45° to 60° E. Present mining operations indicate that the west wall dips at approximately 45° W. At a depth of 35 ft below surface the dips of both walls are flattening, suggesting greater widths than revealed at the surface.

Spodumene occurs in zone 3 and the outer part of the quartz core. Weathering has destroyed most of the spodumene within 20 ft of surface. Spodumene crystals along the outer part of the quartz core are especially massive, often measuring 2 ft wide and 6 in. thick, with a length of more than 5 ft exposed. The walls of the gopher holes sunk in the floor of the main pits contain as high as 70 pct spodumene, 40 pct being common. Width of the spodumene zone varies from 30 ft (15 ft on each side of the core) to 60 ft. Although current erratic mining

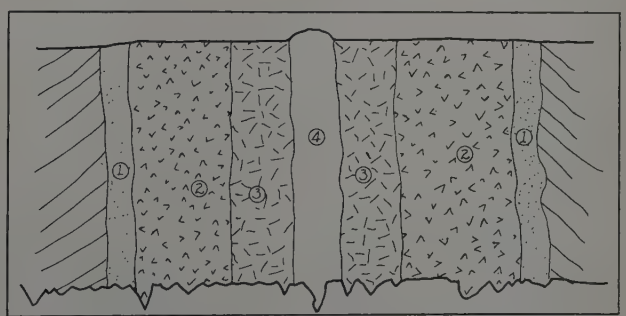


Fig. 3—A cross-section of zoned pegmatite. Circled numbers indicate the following zones: 1—a fine-grained contact; 2—normal pegmatite complex; 3—massive feldspar; and 4—the quartz core.

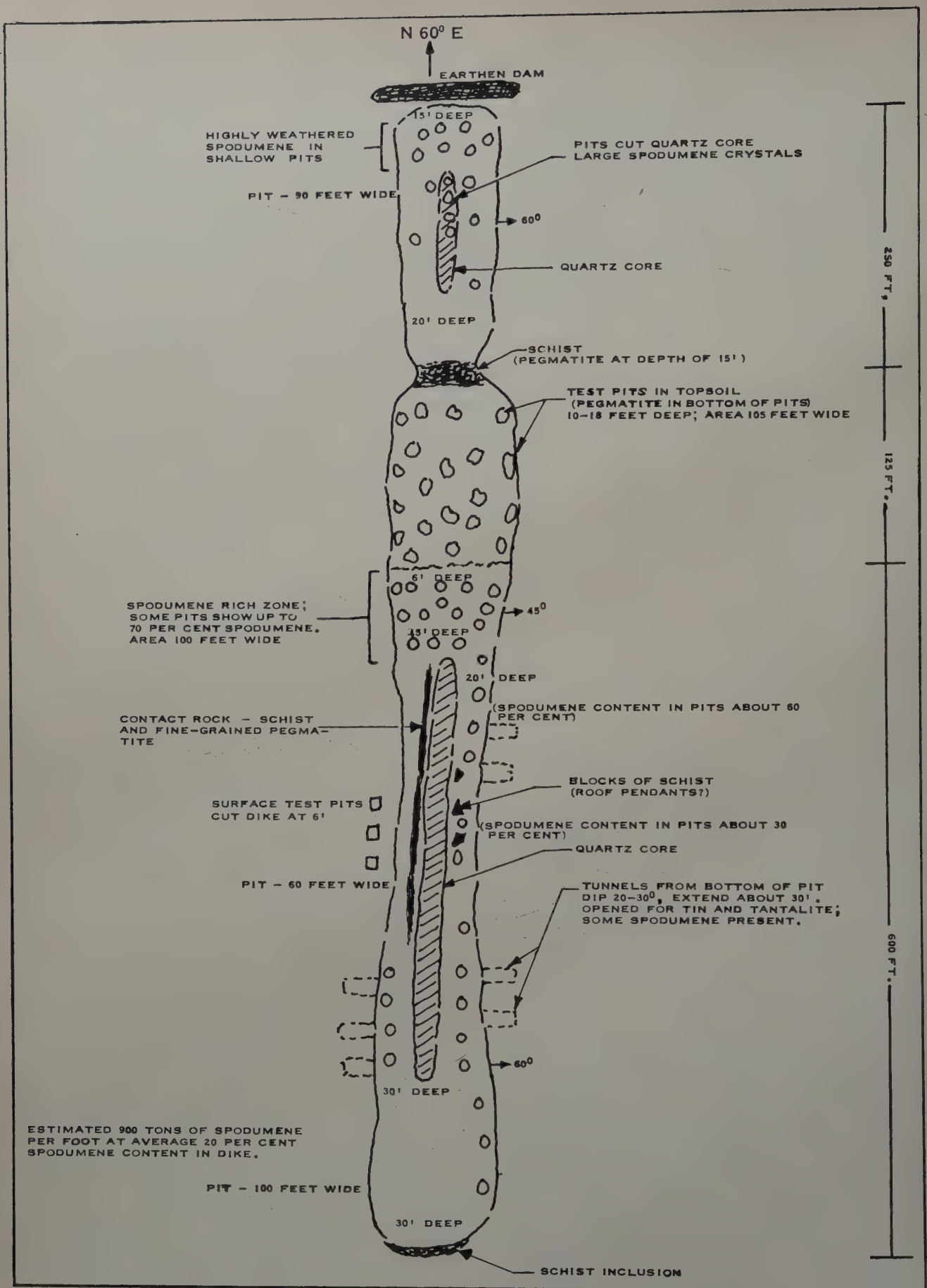

N 60° E
EARTHEN DAM
15' DEEP
HIGHLY WEATHERED SPODUMENE IN SHALLOW PITS
PITS CUT QUARTZ CORE LARGE SPODUMENE CRYSTALS
PIT – 90 FEET WIDE
60°
QUARTZ CORE
20' DEEP
SCHIST (PEGMATITE AT DEPTH OF 15')
TEST PITS IN TOPSOIL (PEGMATITE IN BOTTOM OF PITS) 10–18 FEET DEEP; AREA 105 FEET WIDE
250 FT.
125 FT.
6' DEEP
SPODUMENE RICH ZONE; SOME PITS SHOW UP TO 70 PER CENT SPODUMENE. AREA 100 FEET WIDE
45°
15' DEEP
20' DEEP
(SPODUMENE CONTENT IN PITS ABOUT 60 PER CENT)
CONTACT ROCK – SCHIST AND FINE-GRAINED PEGMATITE
BLOCKS OF SCHIST (ROOF PENDANTS?)
SURFACE TEST PITS CUT DIKE AT 6'
(SPODUMENE CONTENT IN PITS ABOUT 30 PER CENT)
QUARTZ CORE
PIT – 60 FEET WIDE
TUNNELS FROM BOTTOM OF PIT DIP 20–30°, EXTEND ABOUT 30'. OPENED FOR TIN AND TANTALITE; SOME SPODUMENE PRESENT.
600 FT.
60°
30' DEEP
ESTIMATED 900 TONS OF SPODUMENE PER FOOT AT AVERAGE 20 PER CENT SPODUMENE CONTENT IN DIKE.
PIT – 100 FEET WIDE
30' DEEP
SCHIST INCLUSION

Fig. 4—Sketch of the Seridozinho workings.

practice makes ore estimations hazardous, it is believed that the average spodumene content in zones 3 and 4 is in excess of 20 pct; therefore the mine could produce at least 900 tons of spodumene per foot of depth. The owner was asking 40 centavos per kilo ($8.00 per ton) for hand-sorted spodumene.*

* The mine was revisited by the author in June 1954. The owner stated that during the past few months she had sold 800 tons of spodumene concentrates to buyers from Campina Grande and Recife for 1.80 cruzeiros per kilo ($36.00 per ton at the mine). The mine is now closed because the owner is demanding 2.50 cruzeiros per kilo ($50.00 per ton at the mine).

The possibilities for production of spodumene, tantalite, cassiterite, and beryl from this pegmatite are attractive. The dike has adequate length and is widening with depth. Unmined roof pendants, observed at ground level, indicate that the present land surface may coincide with the upper portion of the dike. The vertical extent of the dike may therefore be considerable.

Pedras Pretas: This inactive mine, 3 miles northeast of Seridozinho mine and 9 miles northwest of the town of Juazerinho, was a cassiterite producer. A pegmatite 4 to 10 ft wide was mined by open pit along the strike for 300 ft to a maximum depth of 40 ft. The strike is N 45° E; the dip varies from vertical to 75° E. The wall rock is mica schist.

Subsequent mining efforts were located at the northeast end of the narrow cassiterite producer where a roughly circular pit was opened over a diameter of 200 ft to a maximum depth of 25 ft. The rock in the floor and walls of the pit is a mixture of blocks of country rock and zoned pegmatite. A small area of the massive quartz core is also exposed in the floor of the pit. Spodumene was found in the massive feldspar of zone 3, while spodumene casts were observed in the quartz core. Weathering has highly altered the exposed spodumene.

A third area, immediately to the northeast of the circular pit, has been investigated by a smaller excavation 60x50x25 ft. Identical spodumene-bearing material was found.

The latter two excavations suggest a total dike length of at least 300 ft. The spodumene content, based on present exposures, is lower than that of Seridozinho.

Piaba: The deposit is located 4 air miles, or 11 miles by road, northwest of Pedra Lavrada. At present beryl, tantalite-columbite and spodumene are being mined from an irregular-shaped open pit, 150x75x35 to 50 ft deep.

The pit is located in the southern part of a pod-shaped pegmatitic mass with the long axis striking north-south. The dike, 100 ft wide, crops out for 250 ft along the strike. Country rock is mica schist.

The pegmatite is erratically zoned with large spodumene pods occurring in the feldspar of zone 3 and also in the quartz core. The spodumene is often associated with fine-grained light green mica and rose quartz. Spodumene content of the south wall was estimated at 40 pct.

Stockpiles contained about 100 tons of hand-sorted spodumene, for which the owner was asking 40 centavos per kilo ($8.00 per ton). Current production was at the rate of 8 tons of hand-picked spodumene per week.

Serra Branca: This pegmatite[9-11] is located 12 miles, by road, southeast of Pedra Lavrada, or 14 air miles southeast of Piaba mine. During World War II it was mined for beryl and tantalite-columbite. According to the owner, beryl crystals as large as 50 tons have been mined. There has also been a minor production of cassiterite and amblygonite.

The pod-shaped pegmatite has been explored and mined by an open pit 300x100x50 ft deep. The long axis strikes northwest; the dip is vertical. The country rock is mica schist. Irregular-shaped masses of white to greenish amblygonite occur erratically in microcline and orthoclase of zones 3 and 2. Although individual amblygonite masses measure 2 ft in cross-section the overall percentage is low. Black tourmaline and arrojadite are conspicuous components of zone 2. Cassiterite was observed intergrown in masses of fine-grained greenish mica.

Conclusion: Seridozinho offers the most promising known lithium deposit in northeast Brazil. Pedras Pretas requires drilling to determine the extent of the spodumene zone. This particular area merits development, as it probably contains other spodumene deposits. Because of overburden, exploration for pegmatites can best be accomplished by geophysical methods. In the Piaba and Serra Branca areas many large unexplored pegmatites crop out. Since spodumene weathers to a depth of at least 20 ft, these pegmatites must be prospected by drilling or test pitting.

Deposits in the State of Rio Grande Do Norte

Of the 35 pegmatites examined in Rio Grande do Norte, see Fig. 3, seven contain lithium minerals. Ridges extending for several hundred feet above the surrounding valleys are the principal topographic features. Because of very low rainfall and lack of vegetation and topsoil, the area is easy to prospect.

Boquerao: The pegmatite[12] is located on a ridge top about 900 ft above and two miles east of the town of Parelhas. The deposit, now inactive, was mined during World War II as a tantalite-beryl producer. The owner stated that 300 tons of beryl and 25 tons of tantalite-columbite had been produced. Open pit mining has exposed the pegmatite for 600x100x35-ft maximum depth. The quartz core has been circumvented, leaving this large mass, 300x30 x25 ft high, standing along the central axis of the dike, see Fig. 6.

The pegmatite is vertical. It strikes N 60° W and intrudes a conglomerate that strikes N 80° W. The principal lithium mineral is spodumene. Some lepidolite and amblygonite were noted. The spodumene zone is 6 to 8 ft wide on the south side of the core and 3 to 10 ft wide on the north side. Spodumene crystals in the quartz core are very coarse. Weathering has removed part of the spodumene; its original presence is revealed by casts in massive quartz.

The spodumene content is difficult to estimate because the floor of the pit is partially concealed by an accumulation of debris. The owner stated that the spodumene content was about 10 pct.

Mulungu: This pegmatite has recently been opened as a beryl and tantalite-columbite producer. It is located 4½ miles southeast of Parelhas, along the side of a ridge at an elevation of about 600 ft above the surrounding country. The only means of access is on foot.

The strike is N 70° W and the dip vertical. It intrudes the same conglomerate as Boquerao. The dike has been exposed by a series of small open pits 15 to 25 ft deep along the strike for a distance of 400 ft. The width is unknown, but it is believed to be less than 40 ft.

Spodumene, encountered in the largest pit at the northwest end of the workings, occurs in

Fig. 5—Spodumene stockpiles at the Seridozinho mine.

massive quartz (zone 4) along a 20-ft face. The spodumene content was estimated at 5 to 10 pct. Similar occurrences were observed in the smaller pits to the southeast. Amblygonite intergrown with feldspar and white beryl was noted in one pit. The pegmatite was not sufficiently exposed to permit an estimate of available spodumene.

Fazenda Velha: This deposit,[12] located 6 miles east, southeast of Parelhas, was mined during World War II for beryl and tantalite. A production of 300 tons of beryl and 12 tons of tantalite-columbite was reported. It has been virtually inactive since then.

The pegmatite is vertical, striking northeast and intruding mica schist. It has been exposed along the strike by an ellipitical-shaped open pit 150x45x50 ft deep. A section of the quartz core has been left standing along the central axis of the dike.

Spodumene occurs over a width of 8 to 10 ft in zone 3, on each side of the quartz core, for a distance of 60 ft along the strike. The accumulation of debris on the floor of the pit prevented a detailed examination of the spodumene zone.

Malacacheta No. 1: This deposit is located 5.2 miles south of Parelhas. The pegmatite, which strikes N 70° E, has been exposed by five pits for 200 ft along the strike. The largest of these is 15 ft in diam and 60 ft deep. The wall rock is mica schist. Although zoning is not apparent, it is believed that the present operations are in zone 2. A small quantity of amblygonite was identified in the largest pit. Mica is the only mineral that has been produced.

Sao Sebastiao: This dike is identical with Malacacheta No. 1 and is also a mica producer. It is located 0.8 miles to the west of Malacacheta No. 1. The deposit is mined by two pits, 60x30x25 ft and 50x20x25 ft. A small quantity of amblygonite was identified.

Marimbondo: The mine, now inactive, was formerly worked for beryl, tantalite, and cassiterite. It is located 2½ miles S 60° W of Carnauba. The pegmatite is elliptical in outline and crops out on the flank of a ridge about 300 ft above the surrounding valley. It intrudes a mica schist. Open-pit mining was conducted to a depth of 45 ft, but the quartz core, 75x20 ft, was left standing. Several unexplored pegmatites were noted along the same ridge.

Biotite is especially abundant in zone 1, and there is an unusually large amount of muscovite in zones 2 and 3. The spodumene-bearing area, zone 3 and the outer part of the core, averages about 15 ft wide. The spodumene occurrences in the quartz core are massive crystals, 7 to 8 ft long; crystals weighing over 1000 lb are reported to have been mined. One face of the pit, 35x20 ft, contains more than 15 pct spodumene. A second face, 20x10 ft, carries an estimated 30 pct spodumene. The floor of the principal spodumene zone (zone 3) is partially covered by debris, preventing a more complete estimate of the spodumene content.

Brauna: This inactive mine is located 21 miles north of Parelhas on the Fazenda of Possidonio Avelino de Medeiros. It can be reached only on horseback or on foot. A shallow pit has exposed a zoned pegmatite at least 120 ft long and 45 ft wide. The country rock is mica schist. Beryl, tantalite-columbite, and amblygonite occur in small quantities in zone 3. The owner claims to have sold 10 tons of amblygonite for 40 centavos per kilo ($8.00 per ton). If so there must be richer amblygonite-bearing dikes than the Brauna somewhere on the fazenda.

Conclusion: The extent of the spodumene zone at Boquerao, Fazenda Velha, Mulungu, and Marimbondo must be determined by drilling before their productive capacity can be estimated.

Since five lithium deposits are known in the Parelhas district, it is reasonable to expect other occurrences among the many large unexplored pegmatites in the same district.

The most promising prospecting district, however, is believed to be in the vicinity of Carnauba, especially in the area represented by the Marimbondo pegmatite. Many larger unexplored pegmatites were observed in this vicinity. It is highly improbable that all the lithium was concentrated in the one deposit (Marimbondo).

Deposits in the State of Ceara

Fifteen pegmatites were examined in Ceara. Four contained lithium minerals. Johnston has described[13] six other amblygonite-bearing pegmatites in the Quixeramobim-Cachoeira area and four in the Cascavel-Cristais area. The district is one of deeper topsoil, and prospecting for pegmatites is therefore more difficult than in most of neighboring Rio Grande do Norte and Paraiba.

Soledade No. 2: This mine is located 4.5 miles northwest of Solonopolis. It is a beryl-tantalite producer. Minor amounts of spodumene and amblygonite have also been mined. The zoned dike is over 300 ft long; the width varies from 20 ft at the southwest to 60 ft at the northeast. It strikes N 75° E, discordantly to the enclosing micaceous quartzite. The dip is vertical.

A lenticular-shaped open pit has exposed the pegmatite for 300 ft along the strike and to a depth of 25 ft. The spodumene zone is exposed for 300 ft along the strike and ranges in width from 1.5 to 30 ft. Spodumene crystals are smaller in the coarse-grained feldspar of zone 3 and larger in the outer part of the quartz core. It has been partially weathered to a depth of at least 25 ft. The spodumene content is about 8 to 10 pct. Amblygonite occurs in zones 3 and 2, southwest of the main quartz core outcrop. Beryl and tantalite occur further to the southwest in zones 2 and 3.

Soledade No. 1: This deposit is located one mile south and east of Soledade, No. 2. A narrow east-west trench in topsoil (maximum width of 20 ft) has partially exposed a narrow pegmatite for 100 ft along the strike. The latter intrudes micaceous quartzite at a small angle. Isolated masses of amblygonite were identified. Small quantities of beryl and tantalite have been mined.

Soledade No. 4: This dike is located about 1.5 miles north of Soledade No. 2. A pit 200x20x5 ft has exposed a vertical pegmatite striking N 80° E, discordant to the micaceous quartzite country rock. It had been mined for beryl. Amblygonite, associated with fine-grained greenish mica, was observed in massive feldspar in the east part of the pit. The quartz core was not exposed.

Auriverde: This mine is located 12 miles north of the town of Solonopolis. The long axis of the pit, and presumably of the pegmatite, strikes N 60° W. The country rock is mica schist. The dike is exposed in an open pit 200x40x40 ft deep. Currently it is mined for tantalite and beryl by sinking gopher holes in the floor of the main open pit. A small quantity of amblygonite is produced. A few spodumene and small blue apatite crystals were noted.

Conclusion: Of the four pegmatites examined on the Soledade property, three carried lithium minerals. One of these, Soledade No. 2, by employing modern mining and beneficiation practice, could probably become a producer. The lithium content is too low and the distribution erratic for economic development in the other three lithium-bearing pegmatites. Johnston's description of other amblygonite-bearing dikes in Quixeramobim-Cachoeira and Cascavel-Cristais areas does not offer much encouragement for lithium production from the pegmatites of these areas.

Mining is carried on by primitive hand methods. Dynamite or black powder is used sparingly in a few instances in the state of Ceara.

The pegmatite minerals are concentrated by hand-picking *in situ* and carried from the pits in baskets on the backs or heads of the garimparoes (miners). The tin and tantalite concentrates are stockpiled in adobe or thatched sheds. In a few cases, lithium minerals are stockpiled beside the open pits. On the average, garimparoes receive 25 cruzeiros per day ($0.50 at present rate of exchange). The cost of trucking spodumene 250 km is estimated at 350 to 450 cruzeiros per ton ($7.00 to $9.00 at present rate of exchange).

Spodumene and amblygonite concentrates at the mine were offered at 40 centavos per kilo, or 400 cruzeiros per ton ($8.00 at current rate of exchange). Concentrates could, therefore, be deposited at the ports of Natal or Recife for about 750 to 850 cruzeiros or $15.00 to $17.00 per ton, depending on the existing rate of exchange.

The Brazilian government export quota on lithium concentrates for 1953 was 3000 tons. Fortunately the exportation of lithium minerals is under the jurisdiction of the Commission for Exportation of Strategic Materials, rather than the usual government agency. At present there is no quota on the exportation of lithium salts. To overcome the export restrictions on ore, it would appear logical to convert the concentrates to lithium salts in Brazil.

Fig. 6—Quartz core isolated by mining of massive feldspar. Unmined zone 2 (Boquerao) adjoins feldspar.

General Conclusions

The pegmatites of northeast Brazil, particularly in the states of Rio Grande do Norte and Paraiba, offer possibilities as sources of lithium ore. Lithium minerals were found only in zoned pegmatites.

As sources of lithium ore, Brazilian spodumene deposits are considered preferable to amblygonite deposits. Spodumene crystals occur in greater concentrations; they are more uniform in distribution; and they are more easily separated from the enclosing quartz, feldspar, or mica. Spodumene weathers more rapidly than the other pegmatite minerals, whereas amblygonite is one of the most resistant to weathering. Spodumene is usually weathered to a depth of at least 20 ft.

In certain instances beryl, cassiterite, and tantalite-columbite may be produced with spodumene.

Of the 61 pegmatites examined, Seridozinho and possibly Piaba and Soledade No. 2 are potential spodumene mines. Pedras Pretas, Boquerao, Fazenda Velha, Mulungu, and Marimbondo require drilling to determine the extent of their spodumene zones. Unexplored pegmatites in the vicinity of Parelhas and Carnauba in Rio Grande do Norte, Piaba, and Serra Branca in Paraiba merit exploration. Because of heavier overburden, geophysical prospecting methods should be employed in the Seridozinho and Soledade areas.

The choice of beneficiation methods will be limited by the extreme scarcity of water in Rio Grande do Norte and Paraiba.

To avoid export restrictions, some consideration should be given to the conversion, in Brazil, of lithium concentrates to lithium salts.

References

[1] Roderic Crandall: Geographia, Geologica, Supprimento da Aqua, Transporte, é Acudagem nos Estados da Paraiba, Rio Grande do Norte e Ceara. Inspectoria Federal de Obras Contras as Secas Pub. 4 (1910) 137 pp.

[2] L. J. de Morais: Serras e Montanhas do Nordeste. Inspectoria Federal de Obras Contra as Secas Pub. **58** (1924) 2 vol., 242 pp.

[3] P.A.M. de Almeida Rolff: Geologia da Provincia Tantalo Glucinifera da Borborema. Departamento Nacional da Producao Mineral *Boletim 73* (1945).

[4] P.A.M. de Almeida Rolff: Os Pegmatitos da Borborema. *Revista de Escola de Minas* (Oct. 12, 1951).

[5] E. N. Cameron et al: Internal Structure of Granitic Pegmatites. *Economic Geology* (1949) Monograph 2, p. 16.

[6] Ref. 5, p. 20.

[7] W. D. Johnston, Jr.: *Beryl-Tantalite Pegmatites of Northeastern Brazil.* GSA Bull. (1945) **56**, pp. 1036-1039.

[8] Ref. 7, pp. 1065-1068.

[9] P.A.M. de Almeida Rolff: Minerais dos Pegmatitos da Borborema. *Bol. 78,* D.N.P.M. (1946).

[10] P.A.M. de Almeida Rolff: Reservas Minerais do Municipio de Picui. *Bol. 80,* D.N.P.M. (1946).

[11] Sandoval Carneiro de Almeida: Tantalita é Berito em "Alto" Feioe Serra Branca. Avulso 73, D.N.P.M. (1946).

[12] James E. Moore: Lavra de Pegmatitos Tantaliferos é Gluciniferos no Nordeste do Brazil. *Bol. 71,* D.N.P.M. (1945).

[13] Ref. 7, pp. 1060-1068.

Origin of Uranium Deposits

A Progress Report

by Donald L. Everhart

The search for new deposits raises two important questions: Where did the metallic ions that formed the orebodies come from? What processes and geologic factors were involved in ore replacement? A review of the wide geological variety of recent uranium discoveries tells how these questions are being answered.

SOONER or later intelligent exploration for uranium leads to these questions: Where did the metallic ions that formed the orebodies come from? What processes and geologic factors were involved in ore replacement? These matters, of course, make a big difference in how best to look for orebodies.

Before these questions are discussed it might be well to point out other factors bearing on the subject. The whole business of uranium exploration is only a little over ten years old, participation by the public is only about six years old, and real interest and activity by more than a few large mining companies has arisen only in the past two or three years. Throughout this short period, uranium exploration has been affected by the unique influences of necessary security measures and by division of the overall job into individual projects by a large number of specialists in diverse fields. This has meant that a normal interchange of information on the nature of uranium deposits has really only just begun. Compared to the vast amount of published studies on iron, copper, lead, zinc, and gold deposits over the past 50 to 75 years, current knowledge of uranium deposits is in its infancy and much of the pertinent information is still scattered about in a number of unpublished sources. The volume of reports on all phases of uranium exploration is now beginning to mount at a remarkable rate, and geologists with widely varying specialized backgrounds in uranium geology are engaging in spirited debates over the origin of uranium and the relative importance of geologic controls of various deposits.

One of the most interesting features of known uranium deposits of the world is the wide variation, see Table I, in character and geologic environment. It should be noted that many types became prominent during 1953.

The davidite-bearing veins of **Radium Hill, South Australia,** are almost unique, although the same mineral occurs in a different kind of deposit in Mozambique. Recent studies indicate that brannerite disseminated in the granite near **Crocker's Well, South Australia,** may soon constitute uranium ore. Basalt has been added to the list of favorable host rocks in the **Northern Territory of Australia.** Basalt is also the host of the pitchblende veins of the Martin Lake deposit, **Beaverlodge district, Saskatchewan.** The truly remarkable rich and extensive Steen deposit near **Moab, Utah,** is of the uraninite-vanadium oxide type. Secondary hydrous uranium arsenates and phosphates constitute the uranium minerals in the disseminated deposits of the **Wind River Basin, Wyo.,** a new district discovered in the summer of 1953. Uranium minerals in shales adjacent to fractures were newly reported in New Mexico and several other parts of the world during the year. The well-known important uraninite dissemination in the Rand conglomerates of South Africa has a counterpart, as discovered in the summer of 1953, in the Mississagi quartzite and conglomerate of the **Blind River district, Ontario.** Gunnar deposit of the **Lake Athabaska district, Saskatchewan,** consists of uraninite in both granite and sedimentary gneiss beneath a granite cap, and large new orebody of the Eldorado Co. in the same general area follows a preferred stratigraphic horizon in schists.

Uranium is, generally speaking, a very soluble and ubiquitous element. It occurs in at least two valency states and for this reason is not easily tied down in natural solutions. As Gruner[1] has pointed out, it is probable that uranium starts in the hexavalent state in nearly all solutions instrumental in its transfer. It is believed that under oxidizing conditions the uranium moves in solution as the uranyl ion, UO^{++}. In solutions ranging in pH from 1 to 5, the uranyl ion is most commonly in association with

D. L. EVERHART is Chief of the Geologic Branch, Division of Raw Materials, U. S. Atomic Energy Commission.

Discussion on this paper, TP 3874I, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript April 12, 1954. New York Meeting, February 1954.

Table I. Classification of Uranium Deposits by Host Rock

HOST ROCKS		FORM OF DEPOSIT	DESCRIPTION OF URANIUM DEPOSITS
IGNEOUS ROCKS	INTRUSIVES	VEIN	URANINITE IN SILICEOUS-PYRITE-GALENA VEINS URANINITE IN NICKEL-COBALT-COPPER-SILVER VEINS *DAVIDITE IN SILICEOUS TITANIUM-RICH VEINS
		PEGMATITE	URANIFEROUS PEGMATITE
		DISSEMINATED	*BRANNERITE IN GRANITE
	EXTRUSIVES	VEIN	*URANINITE IN VEINS CUTTING BASALT URANINITE IN VEINS CUTTING ANDESITE URANINITE IN VEINS CUTTING RHYOLITE
		DISSEMINATED	URANIUM PHOSPHATES IN EXTRUSIVE ROCKS
SEDIMENTARY ROCKS	CLASTIC SEDIMENTS	DISSEMINATED IN FLUVIAL SANDSTONES AND CONGLOMERATES	*URANINITE-VANOXITE DEPOSITS COPPER-URANIUM DEPOSITS URANIFEROUS ASPHALTITE DEPOSITS CARNOTITE DEPOSITS *DEPOSITS OF "SECONDARY" HYDROUS URANIUM OXIDES, PHOSPHATES, ARSENATES, ETC.
		DISSEMINATED IN SHALES	*URANIUM MINERALS IN SHALES ADJACENT TO FRACTURES AND SHEARS URANIFEROUS BLACK SHALES
		PLACERS AND BEACH SANDS	URANIFEROUS PLACERS AND BEACH SANDS
	CHEMICAL-ORGANIC SEDIMENTS	DISSEMINATED IN CALCAREOUS SEDIMENTS	URANIUM MINERALS IN LIMESTONE, ADJACENT TO FRACTURES URANIFEROUS PHOSPHATIC DEPOSITS
		LIGNITE BEDS	URANIFEROUS LIGNITE DEPOSITS
METAMORPHIC ROCKS	METAMORPHOSED SEDIMENTS	VEIN	IN SCHISTS AND PARAGNEISS
		DISSEMINATED	*IN QUARTZITE AND METACONGLOMERATE *IN SCHISTS AND PARAGNEISS
	METAMORPHOSED IGNEOUS ROCKS	VEIN	IN AMPHIBOLITES

*SIGNIFICANT DEVELOPMENTS IN 1953

Table II. Tentative Classification of Uranium Deposits by Origin

	MAGMATIC ORIGIN	HYDROTHERMAL ORIGIN	ULTIMATE ORIGIN OBSCURED; AFFECTED BY GROUNDWATER REDISTRIBUTION	POSSIBLE GROUNDWATER FLUSHING FROM VOLCANICS	SEDIMENTARY ORIGIN	BIOGENIC ORIGIN
DEPOSITS IN IGNEOUS ROCKS	BRANNERITE IN GRANITE	PITCHBLENDE IN VEINS CUTTING FELSIC INTRUSIVES				
	URANIFEROUS PEGMATITES	URANINITE IN VEINS CUTTING EXTRUSIVE ROCKS				
		URANIUM PHOSPHATES IN EXTRUSIVE ROCKS				
DEPOSITS IN METAMORPHIC ROCKS		PITCHBLENDE OR DAVIDITE IN VEINS CUTTING METASEDIMENTS OR AMPHIBOLITES				
		(*)	URANINITE DISSEMINATED IN METASEDIMENTS		(?)	
DEPOSITS IN SEDIMENTARY ROCKS		PITCHBLENDE OR THUCOLITE IN VEINS CUTTING SEDIMENTS				
		(*)	COPPER-URANIUM DEPOSITS		(?)	
		URANIUM MINERALS IN LIMESTONE, ADJACENT TO FRACTURES (?)				
			? URANINITE-VANOXITE DEPOSITS ?			
			? CARNOTITE DEPOSITS ?			
			"SECONDARY" HYDROUS URANIUM OXIDES IN SEDIMENTARY BASINS ?			
			URANIFEROUS ASPHALT DEPOSITS			
				URANIFEROUS LIGNITE DEPOSITS		
					URANIFEROUS BLACK SHALES (?)	
					URANIFEROUS PHOSPHATIC DEPOSITS	
					URANIUM MINERALS IN PLACERS AND BEACH SANDS	

(*) more likely origin
(?) less likely origin

$SO_4^=$, as far as most natural acidic solutions are concerned. Furthermore this solubility is effective down to room temperatures. It is also a well-established fact that several kinds of basic carbonate solutions, with a pH of 8.5 upward, hold uranium as complexed ions, at temperatures mostly from about 150°C upward. Thus only solutions that are nearly neutral, with pH from 5 to 8.5, usually fail to hold uranium in solution. Actually there are very few data available on the solubility of uranium compounds in various natural solvents, and much of the data that are available are in the confidential files of individual researchers. Much more printed information is expected in the next few years.

Although uranium in general is a comparatively soluble element, there are a number of effective precipitants. One of the most spectacular of these is carbonaceous matter, which acts in at least two ways: 1—it creates reducing conditions, during decomposition, with the production of H_2S, thereby radically changing the pH; and 2—it possesses powerful capacity to adsorb uranium from many kinds of solutions. High base-exchange clays also have a high affinity for uranium. In areas where uraniferous solutions have a pH of 5 or less any kind of carbonate acts as a precipitating agent. In the special case of certain pitchblende vein deposits, the botryoidal masses are commonly considered to represent the coagulation of a colloid. This largely depends on the presence of a suitable electrolyte. Thus when uraniferous colloids invade any new environment that produces charges opposite to those on particles in the colloid, neutralization, destruction of the dispersive property of the colloid, and deposition ensue. Emmons[2] also suggests transport of uranium as a halide, particularly in igneous rocks, with precipitation caused by decreasing pressure.

Within the past year the U. S. Geological Survey[3] has started a project to appraise the relative horizontal and vertical transmissivity of all the exposed sedimentary rocks of the Colorado Plateau. By use of water consumption data from four representative drilling projects, the Survey recently has evaluated the effect of fracture systems on the transmissivity of the Salt Wash member of the Morrison formation. Preliminary results show quantitatively a correlation between highly fractured, that is, highly transmissive areas and known ore deposits.

In the vein types of deposits, the hydrothermal alteration work of Kerr[4] and his associates in the Marysvale district, Utah, and that of Wright[5] in the Boulder batholith, Mont., have indicated the power of uraniferous hydrothermal solutions, in places, to soak several tens or even hundreds of feet into the granitic wall rock of the veins. This is reflected by an orderly zonal arrangement of mica and clay minerals, and parallels in a general way the observations on hydrothermal effects in the Tintic district, Utah, by Lovering[6] and in the Butte district by Sales and Meyer.[7] The same sort of evidence has been observed in Portugal.

In intrusive igneous rocks, as Brown[8] has pointed out, uranium has an ionic radius and charge which prevents it from fitting comfortably into the lattices of any of the common major granitic minerals, with the possible exception of biotite, but it can fit fairly well into the lattices of certain accessory minerals, notably zircon, sphene, pyrochlore, allanite, and apatite. Therefore, uranium appears to be generally localized in igneous rocks in those minerals which make up only a small fraction of the total weight of the rock. In studies of the distribution of uranium in granites, it has been noted that substantial fractions are not localized in the crystal lattices at all but appear to exist interstitially. These fractions are so loosely bound to the minerals that complete removal can be obtained by leaching the pulverized rock with dilute acids.

One more aspect of the geochemistry of uranium is pertinent to the problem of origin. Considering Mason's geochemical cycle, it is obvious from the classification of known uranium deposits, Table II, that uranium shows up in virtually every part of the cycle. Presumably because of its high solubility over a wide range in pH, temperature, and pressure, it can survive many of the geologic processes. Likewise, because of the variety of precipitants of uranium, many geologic environments in the cycle are favorable for precipitation and concentration.

Origin of Uranium Deposits

Putting together the widely varying geologic character of known deposits of uranium and some of these geochemical properties, it is possible to speculate to some extent on the origin of the uranium ions. From the standpoint of origin, see Table II, it may be concluded that the uranium present in accessory minerals of a granite or a pegmatite was formed during crystallization of the magma. This idea is commensurate with the principles of Bowen and other accepted ideas of magma cooling. Elsewhere in the geochemical cycle, it is almost equally clear that uranium held in organic compounds in such shales as the Chattanooga, which is more or less consistent in grade and character over hundreds of square miles, was laid down by the established processes of marine sedimentation. Uranium in the phosphate beds of Florida is also apparently part of the marine sedimentary rock itself, although in both the shales and phosphates redistribution in varying degree has undoubtedly taken place. The sedimentary nature of such minerals as detrital euxenite or fergusonite in placers is clear-cut.

Almost as easy to prove is the hydrothermal origin of typical uranium-bearing veins in various types of bedrock. The close association of pitchblende with Bastin's[9] nickel-cobalt-silver type of mesothermal vein deposits has been commonly observed. Many of the other uraniferous veins are of the relatively low-temperature silica-pyrite-chalcopyrite-galena type so common in hydrothermally formed mineral districts. In both types of deposits pitchblende occupies a variable paragenetic position, but its close genetic association to other hydrothermal minerals is well established.

The greatest doubts as to the origin of uranium deposits concern the disseminated deposits in sedimentary rocks. Foremost among these, of course, are the bedded deposits in the Colorado Plateau.

Most of the Colorado Plateau deposits are closely associated with certain sedimentary features. Ore lies essentially parallel to the bedding, and most of the deposits occur in the thicker parts and commonly near the base of sandstone lenses, many of which represent old scours or channelways; the trend of the long direction of the deposits and the trend of the ore rolls in the sandstone are roughly parallel to the trend of the fossil logs in the sandstone and to the general dip of the crossbedding. These relations suggest that primary structures in the sediments were instrumental in localizing most of the ore deposits. However, within the past year

or two it has become increasingly evident, as pointed out by Rasor,[10] that below the zone of oxidation much of the ore consists chiefly of oxides—uraninite and low-valent oxides of vanadium—and varying quantities of sulphides such as pyrite, bornite, galena, and chalcopyrite. In some ores a hard variety of uraninite previously reported only from hydrothermal vein deposits has been found. Studies by Stieff and Stern[11] of lead-uranium ratios in ores from the Colorado Plateau indicate that regardless of where they occur all the ores are about the same age and are no older than late Cretaceous. In several areas field relations strongly suggest that the deposits may be genetically related to faults, fractures, or salt dome structures. Two main hypotheses are generally advanced to explain the origin of the Colorado Plateau ores. One is that the ores are penesyngenetic and were formed soon after the enclosing rocks were deposited. Subsequently ground water may have dissolved and reprecipitated the ore, but the original uranium is said to have been already present in the host rocks. This hypothesis, of course, provides a reasonable explanation for the relation of ores to sedimentary features, but it still leaves unanswered the discrepancy between the age of the uranium and the age of the enclosing rock (if these ages are valid) and the fact that uranium occurrences are known in at least 18 separate stratigraphic units. A second hypothesis alleges that the deposits are essentially telethermal, somewhat similar in origin to the Mississippi valley lead-zinc ores. It is believed that the ore-bearing solutions originated at depth from an igneous source and ascended along fractures. After these solutions mingled to a great degree with circulating ground waters they must have cooled so that they were no longer strictly *hydrothermal.* The minerals were precipitated in favorable beds and sedimentary traps as much as several miles from the feeder structures.

The difficulties with this second theory are mainly location of the hypothetical igneous source and proof of the connection between major fractures and faults and the ore deposits. It has also been pointed out, particularly by Gruner,[12] that there is meager evidence for solutions having passed through much of the barren rock that necessarily would have been in the path of ore-bearing solutions on the way from feeder conduits to their final deposition sites. Another possible objection is that the very wide horizontal distribution of orebodies in the Colorado Plateau is considered by some to place a strain on entirely hydrothermal sources.

In metamorphosed and granitized sediments, as at the Gunnar gold property or the new Verna deposits in the Beaverlodge district, Saskatchewan disseminated uraninite also follows the bedding and other sedimentary features, but it is near major faults along which many hydrothermal vein deposits occur elsewhere in the district.

Denson, Bachman, and Zeller[13] have added another concept in suggesting that in the case of lignites overlain by even slightly uraniferous volcanics, the downward flushing of ground water through the volcanics may have picked up uranium in solution, which precipitated or adsorbed below by the carbonaceous matter. A similar hypothesis has been advanced by Bain[14] in explaining the uranium deposits in the Todilto limestone of the Grants district in New Mexico. Here ground water flushing of overlying uraniferous sandstone and precipitation by the carbonate rock below is envisioned. These ideas encounter debate in explaining the localization of the deposits, however, and fluorite, which is almost universally associated with the uranium in the Todilto limestone, must be explained this way, especially since fluorite veins in underlying pre-Cambrian rocks are parallel to the elongation of the uranium orebodies.

Nearly all the disseminated deposits in sedimentary rocks appear to have been greatly affected or redistributed to an unknown extent by the action of slightly acidic or slightly alkaline ground water. This is the real source of the dilemma. Since this reworking appears to have been so widespread and pervasive, it may be a long time before ultimate origin is proved in many cases. A few deep drillholes on suspected feeder structures in the Colorado Plateau might answer many questions, and the Atomic Energy Commission is considering plans for such testing. In the meantime, all geologic factors must be considered in relation to uranium deposits of all types. Uranium, after all, is not greatly different in its geochemical behavior from many other metals except that its high solubility causes ore controls to be extremely subtle.

References

[1] John W. Gruner et al: Annual Report for July 1, 1952 to March 31, 1953 (U. S. Atomic Energy Commission Report RME-3044, April 1953).

[2] R. C. Emmons et al: Mineralogic Studies of the Genesis of Uranium in Granite. University of Wisconsin Research Progress Report: *New Methods for Uranium Exploration and Recovery from Low-Grade Ores, May 1, 1951 to January 1, 1952.*

[3] U. S. Geological Survey: *Geologic Investigations of Radioactive Deposits. Semi-annual Progress Report June 1 to November 30, 1953.* (U. S. Geol. Survey Trace Elements *R.I. 390* December 1953).

[4] Paul F. Kerr et al: Annual Report for June 30, 1952 to April 1, 1953 (U. S. Atomic Energy Commission RME-3046, April 1953).

[5] H. D. Wright et al: *An Investigation of the Mineralogy of Uranium-Bearing Deposits in the Boulder Batholith, Montana.* Progress Report No. 4, April 1-Sept. 30, 1953. The Pennsylvania State University, School of Mineral Industries, Oct. 1, 1953.

[6] T. S. Lovering: Rock Alteration as a Guide to Ore—East Tintic District, Utah. *Economic Geology* Monograph No. 1 (1949) 64 pp.

[7] R. H. Sales and Charles Meyer: Interpretation of Wall-Rock Alteration at Butte, Mont. *Colorado School of Mines Quarterly* (January 1950) **45**, No. 1B, pp. 261-273.

[8] Harrison Brown: The Possibilities of Securing Long-Range Supplies of Uranium, Thorium, and Other Substances from Igneous Rocks. California Institute of Technology, 1953.

[9] Edson S. Bastin: The Nickel-Cobalt-Native Silver Ore Type. *Economic Geology* (1950) **45**, No. 4, pp. 273-323.

[10] Charles A. Rasor: Uraninite from the Gray Dawn Mine, San Juan Co., Utah. *Science* (July 25, 1952) **116**, No. 3004, pp. 89-90.

[11] L. R. Stieff and T. W. Stern: The Lead-Uranium Ages of Some Uraninite Specimens from Triassic and Jurassic Sedimentary Rocks of the Colorado Plateau. (U. S. Geol. Survey Trace *R.I. 322* April 1953).

[12] John W. Gruner: The Origin of the Uranium Deposits of the Colorado Plateau and Adjacent Regions. *Mines Magazine* (March 1954) pp. 53-56.

[13] N. M. Denson, G. O. Bachman, and H. D. Zeller: Summary of New Information on Uraniferous Lignites in the Dakotas. (U. S. Geol. Survey Trace Elements Memorandum Report 175 (1950). Unpublished.

[14] George W. Bain: Annual Report for July 1, 1952 to June 30, 1953. (U. S. Atomic Energy Commission RME-3039, March 1953).

Underground Electrocarbonization of Coal And Related Hydrocarbons

by T. C. Cheasley,
J. D. Forrester,
and Erich Sarapuu

Electrocarbonization of coal will produce cheap fuel for electric power plants. The complete electrification of industry and domestic power consumption of gas generated as fuel *in situ* is a worthwhile goal for the coal industry. Production of synthetic oils, gasoline, and coal chemicals from coal gas will create additional new markets for the coal industry.

RANKING high in the nation's wealth of natural resources, readily available liquid and gaseous hydrocarbons are nevertheless falling steadily in supply, while industrial requirements for hydrocarbon compounds are greater today than during the peak period of World War II.

Theoretically, several processes will increase the future supply of liquid and gaseous fuels, but the practical application of these methods depends on two controlling factors: 1—that enough carbon-bearing material be available to warrant industrial exploitation, and 2—that the given process be physically, chemically, and economically applicable. With regard to the first factor, the Western Hemisphere is fortunately well supplied, see Table I.

Table I not only shows the extensive occurrence of many fuel sources, but also the wide discrepancy among quantities of fuel substances in various materials. Great quantities of potentially available fuel have not been recovered. Many coal seams and other hydrocarbon-bearing deposits are so situated that production is uneconomical by present-day mining or oilwell pumping methods, and even in many exploitable deposits where the most efficient production methods are used a large proportion of material is never recovered. Industrial production of hydrocarbon liquids and gases must be better balanced, and processes must be developed which will convert coals, oil sand, and oil shales into fabricated commodities.

Among the techniques employed by scientists and engineers to secure fuel masses not economically available at present are several processes of *underground gasification*. These are all concerned with converting raw materials into gaseous products by thermal treatment of source substances while they are still in the ground and therefore require that a subsurface heat (fire) zone be induced to affect pervasively the buried raw fuel deposit. This is possible only where sufficiently high temperature exists and where the fuel material is satisfactorily opened or channelized. Also, to compete successfully with common mining or oilfield practices, a given method must be controllable and must not involve greater operational expense than regular methods. Preferably, it should be less expensive.

Since the close of World War II, England has continued field studies of underground gasification methods. Experiments have also been conducted in Sweden, Estonia, France, Belgium, Italy, and Germany. The U. S. Government has sponsored field studies at Gorgas, Ala., and because gasification *in situ* is vitally important to establishment of the nation's fuel resources, the mining engineering department at the Missouri School of Mines has been actively interested in developing the process of underground electrocarbonization. Research was conceived by Erich Sarapuu, and preliminary experiments in treating bituminous coal were begun at the school in September 1947. The work consisted of laboratory experimentation until June 1948, at which time, through a cooperative agreement with the Sinclair Coal Co. of Missouri, pilot plant studies were instituted at the Tiger mine of the Hume-Sinclair Coal Co. near Hume, Mo., to test the encouraging laboratory findings. This comprehensive field program was pursued continuously until the beginning of 1952.[8-10]

The broad term *underground electrocarbonization* describes a unique gasification process that releases by electricity the desired economic constituents from a raw carbon-bearing material, converting the substance of such deposits into deliverable form and greatly reducing the cost-of-mining factor. Underground electrocarbonization is not subject to the major limitations of many other underground gasifi-

T. C. CHEASLEY, Member AIME, is Vice President, Sinclair Coal Co., Kansas City, Mo. J. D. FORRESTER, Member AIME, is Dean, College of Mines, University of Idaho. E. SARAPUU, Associate Member AIME, is President, Electrotherm Research Corp., Kansas City, Mo.

Discussion on this paper, TP 3873F, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript, Feb. 10, 1954. New York Meeting, February 1954.

cation procedures. That is, the digging of underground drifts is not necessary, and control of the process can be closely maintained throughout the operation. Access to the raw fuel *in situ* is attained by means of vertical boreholes which may be placed several hundred feet apart. Electric current is transferred to the fuel medium by pipeline electrodes inserted in these holes. The pipes serve also as conduits through which the liquid or gaseous materials generated can be conveyed to the surface.

The General Process of Underground Electrocarbonization

In the case of coal and oil shale, valuable materials may be derived from both fixed carbon and volatile matter. Oil sands can be expected to yield commercially only liquid hydrocarbon compounds. Because of these broad differences in the natural modes of occurrence of various sources of mineral fuels, any particular application of the general procedure of underground electrocarbonization will have peculiar special characteristics or phases. These phases, which may exist singly or in combination during an operation of the process, are as follows:

Electrolinking: As noted previously, it is necessary that linkages or channels be induced within the raw fuel medium to institute and maintain the complete conversion by gasification *in situ* of a mineral fuel deposit. When managed properly, short-duration electrical heating, which utilizes conductivity of the fuel deposit, will give relatively rapid development of such linkages. Known as *electrolinking*, this creates a system of fractures and forms a fixed carbon route along which the electric current can move preferentially rather than on a course governed by the electrolytical conductivity of the fuel mass. If this were not so, the temperature rise in the fuel bed would be limited by the evaporating temperature of electrolyte. Electrolinking affords a means of conducting subsequent gasification through a medium such as coal. In the case of oil accumulations in depleted sands, it may afford channels to stimulate the movement of oil to a well bottom.

Electrocarbonizing: Converting a fuel material *in situ* (at least in part) by electrical heating of relatively long duration is known as electrocarbonizing. When volatile constituents of coal such as byproduct tar compounds and ammonia liquors are released by this process, the physical-chemical changes wrought are essentially those occurring in a coke-oven plant. In fact, a buried coke composed of fixed carbon and ash is formed and a gas of 500 to 550 Btu per cu ft is generated from the volatile matter. This valuable gas comprises about 25 pct of the economic potential of the fuel deposit. Like the so-called byproduct tar compounds and ammonia liquors, it can be delivered to the surface.

Electrocarbonizing also forms the linkages, or channels, necessary if the fixed carbon of the coal is to be obtained by subsequent gasification with air, oxygen, or steam. Electrocarbonizing therefore serves two functions: it increases permeability and it yields volatile hydrocarbon compounds. Thus it will leave *in situ* a residue of coke (fixed carbon and ash) as a channelized fuel bed readily amenable to later subsurface gasification treatment or to subsequent exploration by ordinary strip mining methods.

Electrogasifying: The electrogasifying phase of underground electrocarbonization entails simultaneous electrical heating and injecting of air, oxygen, or steam through the fuel medium *in situ*. In reality it is a combined activity whereby volatile hydrocarbons are produced by electrocarbonizing while additional gases are derived simultaneously from the fixed carbon of the deposit by means of a gasifying process that sweeps air, oxygen, or steam through the linkages in the fuel mass.

The electrogasifying procedure will yield a gaseous mixture from the subsurface fuel deposit which is the equivalent of enriched producer gas or water gas. It is apparent that if this phase is carried to completion in the case of any amenable deposit, all the potential fuel constituents will have been liberated and obtained. The noncombustible rock matter or ash, seldom an economic substance, offers no problem of disposal as it has been left buried.

The following procedure for underground electrocarbonization of a coal deposit will aid the reader to grasp the process as a whole. Except for some pertinent modifications, similar treatment is applied to other types of fuel deposits.

Drilling of Boreholes: At least two boreholes are drilled so that the coal seam can be contacted from the surface. Maximum horizontal distance between boreholes during the pilot-plant experimentation at Hume, Mo., was 60 ft. Test data indicate, however, that for commercial purposes separations up to 500 ft or more would be feasible. Usually the depth to which holes must be drilled to reach the fuel deposit is not critical to successful operation of the process.

Table I. Carbon-Bearing Deposits of the Western Hemisphere

Deposits	Millions of Short Tons
Coal, All Ranks[1]	2,120,480
Oil from Oil Sands and Tar Sands	
United States*	113
Canada (Athabasca), minimum**	8,744
Canada (Athabasca), maximum**	26,230
Total minimum	8,857
Total maximum	26,342
Oil from Oil Shale	
United States, Green River formation, northwest Colorado only	
Mahogany ledge†	13,584[4]
500-ft thick measure†	53,250[4]
Brazil, Iraty black shale[5]	87,300‡
Total (using 500-ft measure for United States)	140,550
Oil from Oil Reservoirs	
Crude oil only	
United States[6]	4,155
Canada[7]	297
Mexico[7]	343
Venezuela[7]	1,485
All other countries in western hemisphere	194
Subtotal	6,474
Natural Gas Liquids	
United States[8]	645
Western Canada[8]	8
Subtotal	653
Total Oil from Oil Reservoirs	7,127

* Fifty percent of measured and indicated reserves of bitumen from bituminous sandstone in deposits near Vernal and Sunnyside, Utah; Casmalia, Santa Cruz, and Edna, Calif.; and Uvalde, Texas.[2]

** Fifty percent of estimated reserves in 8000 to 30,000 square miles.[3]

† Partly blocked and inferred oil shale. Resource reserve figures based on 66 pct net recovery from mining and retorting.

‡ Reserve figures based on 66 pct net recovery from mining and retorting.

Insertion of a Pipeline System: Piping is placed in the drillholes to reach from surface to coal seam. During pilot-plant tests boreholes were of 6-in. diam to accommodate the 2½-in. steel pipe used. An electrode or shoe of 4½-in. stainless-steel pipe was connected to the lower end of the tubing of each hole before it was placed in the hole. The pipe system serves as an electrical contact and also conducts the evolved gas to the surface. Necessary connec-

tions, pipelines, and receivers must be established, therefore, in the unit on the surface.

Applying Electrical Power: Electrolinking requires electrical energy of relatively high voltage to begin the desired reactions, but as carbonization continues voltage must be reduced, since the buried fuel deposit assumes new electrical characteristics through progressive changes caused by heating and by linkage attained between boreholes. These variations require installation of multiple-range transformers and related control devices. During the pilot-plant research at Hume, Mo., 2400-v ac was secured from a high-line transmission system and transformed to lower voltage as needed.

Transmission of Compressed Air, Oxygen, or Steam Through the Heated Fuel Mass: The heat generated by electrical resistance is sufficient to start a reaction, and by injecting atmospheric gases or steam down one well pipe and withdrawing or discharging through the other hole of the two-hole system, it is possible to create a progressive gasification through the interconnecting channelways within the seam. At the bottom of the intake pipe the gasifying medium, such as air, reacts with the fixed carbon to yield chiefly carbon dioxide gas. This material then is converted by endothermic reaction to a carbon monoxide gas mixture as it moves through the additional coke of the seam toward the discharge hole where it will be conveyed to the surface. Air, oxygen, or steam injection is continued until the original fuel material has been progressively affected.

Gas Storage and Associated Facilities: Economic disposal of products yielded by an underground electrocarbonization process of industrial dimension will require a surface plant and conveniences.

Underground Electrocarbonization of Coal

Experimental Procedures: Laboratory experimentation was made on a large sample of bituminous coal from the Melbourn seam which occurs on the property of Sinclair Coal Co. at Hume, Mo. The primary purpose of this work was to ascertain basic data as a subsequent guide in pilot-plant research. The following controlling factors were deemed most important:

1—Magnitude of electrical resistivity of the coal. The resistivity defined the size and voltage range of the electric transformer required.

2—Critical current density necessary to breakdown electric resistance of the coal. This was essential for estimating the size of the power unit.

3—The quality and quantity of gas produced by the process. Measurement of gas volume and Btu content was a means of evaluating general economics of the procedure, or in other words, determining the ratio of input-energy to output-energy.

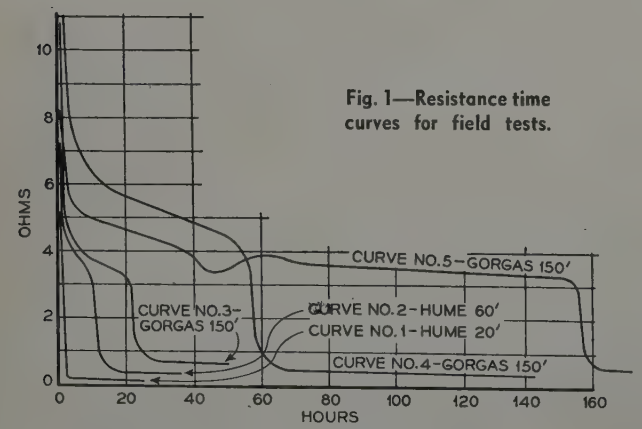


Fig. 1—Resistance time curves for field tests.

Laboratory experiments progressed to successful completion and gas of good Btu value was obtained. It was further demonstrated that under controlled conditions electrolinking could be accomplished in small samples. Also it was shown that the coal could be channelized for subsequent gasification with air, and it was determined that cylindrical electrodes operated with approximately 0.10 to 0.15 amp per sq cm were required to start the process of electrolinking in laboratory samples.

The first field study of underground electrocarbonization was conducted at the Tiger mine of Sinclair Coal Co. where a coal seam lying about 20 ft below the surface was tested. Contact to the coal bed was made by means of insulated electrodes placed in boreholes. Electrical connections were made on the surface to the secondary of a 500-kva, 2400-v transformer. When electric current was applied for approximately 4½ min a very rapid drop in resistance demonstrated that it was possible to duplicate the laboratory tests in the pilot-plant. The tests showed also that voltage control of the secondary of the transformer was absolutely required and that 2400 v was sufficient to accomplish the necessary field operations.

Following the initial success, a second experiment was started with improved equipment. To control the secondary voltage applied to the electrodes, a water rheostat was built. The design of the electrodes was modified so that each could serve not only as a current conductor but also as a gas collector and an air-lift pump. In this second experiment the operation was started by application of a controlled electrical current to electrodes spaced 20 ft apart. The pre-heating or pre-linkage period was about 65 min, during which time resistance in the fuel mass between boreholes was reduced from 7 to approximately 5 ohms. At the end of this time, electrolinking occurred and there was a very sharp drop of resistance to about 2 ohms. The electrocarbonizing phase then was initiated by continued application of electric power, with an accompanying decrease in resistance. This test resulted in production of a very satisfactory gas having a heating value of 525 Btu per cu ft, demonstrating the feasibility of correlating field and laboratory data.

The conclusions reached as a result of the first two field investigations were: 1—A coal deposit can be treated *in situ* by an electrocarbonizing procedure. 2—An electrical potential of 2400 v is satisfactory to initiate electrolinking. 3—Electrolinking in the coal seam will induce channels through which air, oxygen, or steam can be moved and reacted between one borehole and the next. 4—A gasification process by air, oxygen, or steam can be started after the electricity is stopped. 5—A satisfactory gas can be produced by the process of electrocarbonization as a whole.

To extend the scope of further experimental field work beyond the physical limits imposed by use of the water rheostat, a special autotransformer rated at 500 kva with secondary taps down to 200 v was purchased. This transformer, which was capable of giving full kva output down to 800 v, also would deliver 240 kva when operated at 200 v. In addition, several indicating and recording instruments were obtained for measuring electricity and gas. Prohibitive expense during the field tests did not permit

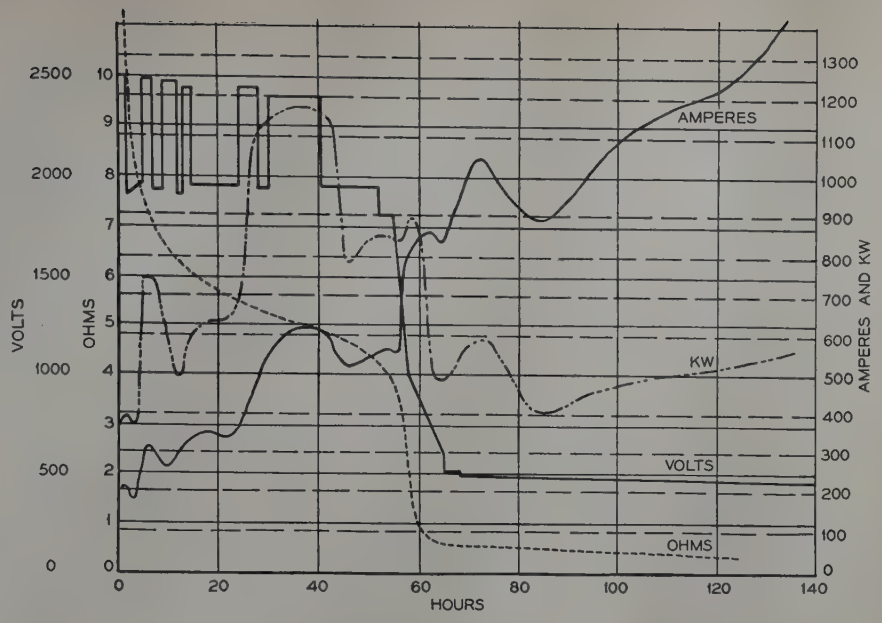


Fig. 2—Power requirements for electrolinkage as related to time curves for voltage, amperage, and kilowatts. Ampere and kilowatt curves follow closely what would be expected from the voltage changes that took place. Electrolinking was accomplished in about 58 hr, after which the electrode potential was reduced to 500 v.

installation of either an electro-filter or a gas exhauster. However, the operation of a full-scale industrial plant, where complete recovery of tar compounds is economically necessary, would require an electro-filter in the system and withdrawal of gas from the subsurface fuel bed by an exhauster.

As a result of continuing experiments at Hume, Mo., successful electrolinking and electrocarbonizing of the coal seam ultimately was obtained with electrode spacing of 60 ft. In this final experiment, resistances decreased from 11 ohms to approximately 7 ohms in a period of 12 hr. At the end of this time electrolinking was accomplished and the resistance dropped very rapidly to less than 0.4 ohm. It continued at this low value throughout the test.

In 1951, Sinclair Coal Co. and the U. S. Bureau of Mines developed an agreement to apply the electrocarbonization process at Gorgas, Ala. Electrical equipment was moved to Gorgas where the process was applied to make a fire-drift or channel in a coal bed previously used for gasification research by the USBM.

The data secured from the Gorgas experimentation have been weighed and analyzed with those obtained earlier at Hume. They have been plotted comparatively on Fig. 1.[10] Curves No. 1 and 2, for data taken at Hume, represent results of tests where the electrodes were spaced at 20 ft and 60 ft respectively. Curves No. 3, 4, and 5 are from records obtained at Gorgas, where in each case the electrodes were 150 ft apart. Of the latter three curves, No. 3, which shows that electrolinkage was accomplished in about 21 hr, or at a rate of about 7 ft per hr, is believed to be the most typical expression of what might be expected from large-scale or industrial operations. This is because the relatively long preheating (pre-linking) time shown in curves No. 4 and 5 is due to the fact that one electrode in each of these tests was in, or very close to, a carbonized area that had resulted from prior tests at Gorgas. Such previously carbonized areas tended to expand the equivalent diameter of the electrode to an extent where current density values were reduced below the point required to penetrate through the coal seam and accomplish electrolinking. These abnormally large electrodes required long times and large kw-hr input to secure sufficient heat through the central path to break down the fuel bed.

Fig. 2 illustrates the power requirements of electrolinking as related to time curves for voltage, amperage, and kilowatts. The ampere and kilowatt curves follow closely what would be expected from the voltage changes that took place. Electrolinking was accomplished in about 58 hr, and then, to maintain a reasonable current on the electrodes, it was necessary to reduce the electrode potential 500 v.

Data and test results indicate that an ideal power source for a full-scale commercial underground electrocarbonization process should be approximately 2000 kva, with a secondary voltage regulation from 4000 to 400 v. Such control should give an optimum current density on the electrodes and a resultant maximum penetration through the coal seam at a minimum power cost to accomplish the electrolinking phase of the process.

Concerted efforts were made to determine the size and shape of the underground gasified area which resulted from the electrocarbonization field research. This was important because a knowledge of the character of the area would give not only a better understanding of the underground gasification technique, in general, but would also guide proper layout and hole distribution of a full-scale, multi-electrode project designed to treat and convert the maximum amount of coal. In as much as the field tests at Hume had been made in shallow coal, it was possible to expose the treated bed by stripping the cover with a dragline and thus ascertain the pattern of the affected zone *in situ*. At Gorgas, where the fuel bed occurs beneath about 200 ft of overburden, it was not economically possible to open the ground for a direct view, and the extent of the gasified area was defined by means of a modified zero potential electrical method. In both cases, at Hume and Gorgas, the pattern of the affected areas was similar to the shape of a crudely center-swollen but attenuated and lengthened ellipse. Fig. 3 illustrates typical gasification patterns developed by uniflow action. In such an operation the air, oxygen, or steam used as a gasifying medium is swept or blown continually in one direction from an intake borehole toward a second or exhaust hole. Thus there is no reversal of direction. Although this method of uniflow motion has produced gas of higher Btu in greater quantities than has ever been done before, analysis of the gas indicates that at

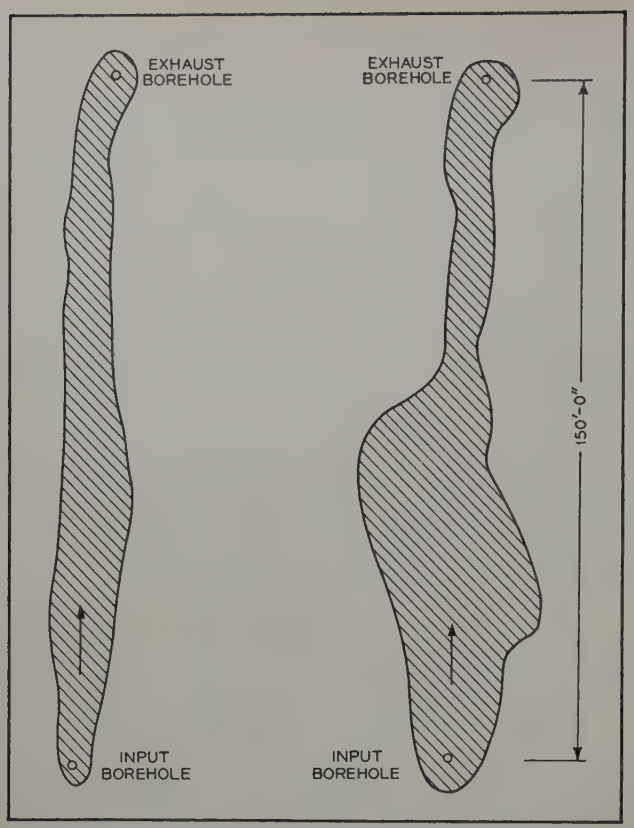


Fig. 3—At left, gasification pattern when oxygen and steam are used in uniflow operation. At right, typical gasification pattern when air is used in uniflow operation.

least 10 pct of the potential carbon dioxide was still not converted into carbon monoxide, as would be the case if it were recycled or passed secondarily over a hot coke bed. Recycling probably could be accomplished if some method of reverse flow were put into effect. Reverse flow also would cause the shape or pattern of the gasified zone to change to a more uniform or symmetrical outline which would increase accordingly the recoverable tonnage of coal lying between any two electrodes. At the present time there are not enough experimental data to permit full comparison between the uniflow and reverse flow methods.

Summary and Economic Aspects

The fundamental advantages of electrocarbonization are: 1—relatively low construction and installation cost, 2—comparatively low cost of operation and maintenance, 3—simple regulation and good control, 4—high efficiency and therefore good economic yield, and 5—handling of noncombustible matter is eliminated.

Though the economics of the process depend on many factors such as cost of power, depth to fuel bed, thickness of seam, and proximity and character of industrial activities, research has demonstrated that underground electrocarbonization of coal probably can be conducted profitably and with success. From the labor standpoint alone, it should create more pleasant and less hazardous jobs and institute new potentialities in the coal industry. The compilation of a cost analysis to illustrate the operating economics of a large-scale commercial plant comprising 30 to 50 underground linkages indicates that it is reasonable to expect production costs of about 10¢ per 1 million Btu.

The growing development of gas turbine installations and the ever increasing demand for electric power may lead to the first commercial adaptation of the process of underground electrocarbonization of coal. For example, the use of low Btu producer gas in turbines already has been studied by both the General Electric Co. and the Westinghouse Electric Corp.:[11]

> On July 29, 1952, an agreement was reached between United States Steel Corporation and Westinghouse Electric Corporation for the erection of a combustion test system at the former's Carrie Furnace of the Homestead District Works. The purpose of this test program was to explore the possibilities of burning blast furnace gas in an internal combustion gas turbine and to develop suitable equipment for such an application.
>
> Initial tests of the combustion design indicated that modifications to the gas admission nozzle were required to improve combustion and flame stability for full and part load operation.
>
> The tests indicated that blast furnace gas can be ignited and burned with efficiencies comparable to those obtained when fuel oils or gases with higher heating values are used in gas turbine combustors.

Operating gas turbine topping units in combination with secondary-load gas turbine systems or low-pressure steam units offers attractive possibilities for generating large blocks of electric power. Generating stations and manufacturing plants, when located in the field at the place of electrocarbonization of raw fuel *in situ*, will inhibit uneconomical movement of gas away from the site by pipeline.

Fig. 4 demonstrates the relative costs of transmitting energy in the form of electricity and coal. It is assumed that 1 ton of coal equals 25 million Btu and 1 kw-hr equals 12,500 Btu. Thus 10¢ gas is equivalent to $2.50 coal. As the costs of transmitting

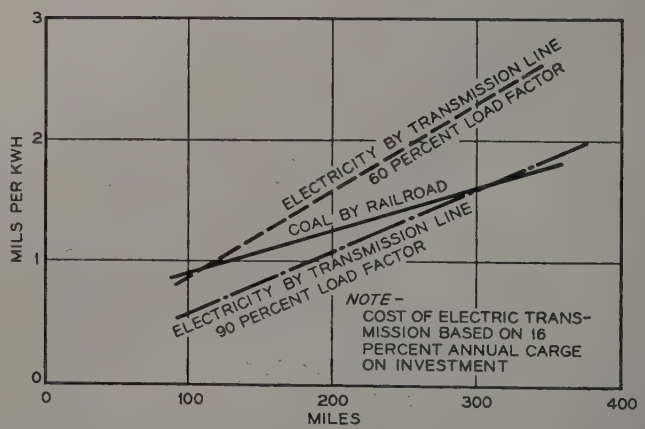


Fig. 4—The relative cost of transporting energy in the form of electricity and coal.

energy are chiefly those of investment, the cost is almost inversely proportional to the load factor and almost directly proportional to the annual charge. The effect of load factor on transmission costs is shown by two curves in Fig. 4; at 60 pct load factor electric transmission is cheaper up to 100 miles, and at 90 pct load factor the curves cross at a distance of approximately 350 miles.

The gasification of a coal seam *in situ* after it had been preliminarily channelized by electrolinking was investigated at Gorgas, Ala., by the USBM. The results obtained by such experimentation have been published by M. F. Fies and J. L. Elder[12] and Tables

II, III, and IV, which follow, have been taken from their publication.

Industrial application of underground electrocarbonization of coal will be determined by the relative cost of the process as expressed in equivalent fuel units and in the comparative availability and character of other competitive types of raw fuel materials.

As mentioned before, oil shale is an important potential source from which petroleum compounds can be derived whenever they are required industrially. The treatment of oil shale *in situ* is an attractive engineering problem; indeed, the general process of underground electrocarbonization first was conceived and applied as a means of securing oil from oil shale, and early research of electrocarbonization was carried out by Erich Sarapuu in the Mining Academy, Freiberg, Germany.[13] Composition of shale used for the first experiments was as follows (Fisher analysis):

Tar	42.10 pct
Moisture	6.80 pct
Fixed carbon	43.30 pct
Gas	7.80 pct
Total	100.00 pct

As is the case in converting buried coal masses by electrocarbonization, the oil shale deposit is prepared for treatment by drilling boreholes into which pipelines are inserted. In general, ground conductivity is a function of the following variables: 1—type of soil, 2—definite anisotrophism of the formation, 3—chemical nature of salts in the electrolyte, 4—temperature of the soil, 5—concentration of salts in the electrolyte, 6—closeness of packing and rock pressure, and 7—moisture content in the rock.

Visual observation in the laboratory of the process of electrolinking of oil shale, using electrode spacing of about 10 cm (4 in.), showed clearly that electrolinking is an electrical activity comprised of two phenomena occurring almost simultaneously, namely, electric arcing and resistance heating. The current, following the least resistance in a rock containing hydrocarbons, creates minute arcs along the path of the current. The fixed carbon which is liberated at arcing temperature forms a fixed carbon zone for resistance heating. The speed of linking is a function of the electric power applied. This cannot be explained as *dielectric puncturing,* wherein the breakdown potential would be proportional to the breakdown distance or voltage gradient (volts per unit distance). Some of the laboratory data showing the characteristic effects of electrode spacing on electrolinking are as follows:

Electrodes Spaced at 10 Cm		Electrodes Spaced at 5 Cm	
v	ma	v	ma
1000	8.5	1000	16
1500	10	1500	25
2000	12	2000	32
2500	13	2500	50
3000	15		
3400	27.5		

These data demonstrate that resistance is affected by electrode spacing. In actual field practice, however, the resistivity changes that transpire over large distances tend to reduce this effect to zero, but good

Table II. Operating Results, Gasification With Air[12]

System	Boreholes XII-XIII	Boreholes XIV-XV	Boreholes SIV-XV
Length, ft	152	155	155
Inlet	Borehole XI or XII	Borehole XIV	Borehole XIV
Outlet	Borehole VIII	System VIII-XII	System VIII-XII
Operating time, days	93	59	21
Air input, cfm*	1195	1160	1890
Gas output, cfm*	692	1171	2157
Input air pressure, psig	8	30	27
Input air leakage, pct	48.1	13.8	5.2
Properties of product gas			
Analysis, pct			
Carbon dioxide	11.9	10.5	9.7
Illuminants	0.3	0.3	0.3
Oxygen	0.5	0.9	0.5
Hydrogen	7.6	8.4	9.2
Carbon monoxide	7.1	10.7	13.1
Methane	1.8	1.8	1.5
Nitrogen	70.8	67.4	65.7
Heating value, Btu per cu ft*	71	86	93
Moisture content, mol water per mol dry gas	0.21	0.15	0.11
Rate of coal utilization** basis of moisture and ash-free coal, tons per day:			
Completely gasified	2.07	5.79	11.15
Carbonized only	8.92	11.29	15.94
Total affected	10.99	17.08	27.09
Total coal utilized** basis of moisture- and ash-free coal, tons:			
Completely gasified	193	342	235
Carbonized only	829	667	329
Total affected	1022	1009	564
Usage and production per ton of moisture- and ash-free coal completely gasified:			
Air used, M cu ft	831	289	243
Gas produced, M cu ft	481	292	278
Heat of combustion of gas, Btu x 10^6	34.2	25.1	25.8
Energy required for air compression,† Btu x 10^6	4.6	4.5	3.6
Net energy produced, Btu x 10^6	29.6	20.6	22.2

* 60°F, 30-in. Hg pressure, dry.
** Calculated from material balances.
† An adiabatic efficiency of 75 pct is assumed for air compression. Power is to be produced from the product gases and it is assumed on conversion to electrical energy, 11,000 Btu will be required per kw-hr.

Table III. Operating Results, Gasification With Oxygen[12]

Operating time, hr	36
Oxygen input rate, cfm*	604
Properties of effluent gas	
Analysis, pct	
Carbon dioxide	47.8
Illuminants	0.3
Oxygen	0.2
Hydrogen	24.5
Carbon monoxide	21.2
Methane	4.1
Nitrogen	1.9
Heating value, Btu per cu ft	195
Specific gravity	1.0
Moisture content, mol of water per mol of dry gas	0.34
Effluent gas flow, cfm*	1335
Cu ft of oxygen per M of make gas	452
Cu ft of oxygen per M of ($CO + H_2$)	990
Cu ft of oxygen per million Btu in the product gas	2320
Rate of coal utilization, basis of moisture-and ash-free coal, tons per day**	
Completely gasified†	33.1
Carbonized only	0

* Measured at 60°F, 30-in. Hg, dry.
** Calculated from material balance.
† This is fresh coal plus coke from previous gasification with air, all calculated as equivalent coal.

Table IV. Water Gas Production, Average Operating Results for 12 Consecutive Cycles[12]

Item	Air Blow	Steam Run
Average length of period, hr	18.8	4.2 (3.85 to 5.08)
Input air rate, cfm*	1250	
Input steam rate, lb per hr		varied from 1200 to 2100
Gas production, cfm*	1800	585
Properties of effluent gas		
Analysis, pct		
Carbon dioxide	12.6	20.8
Illuminants	0.3	0.4
Oxygen	0.3	0.4
Hydrogen	13.4	47.9
Carbon monoxide	10.4	12.0
Methane	2.8	7.6
Nitrogen	60.2	10.9
Heating value, Btu per cu ft	112	279
Specific gravity	0.92	0.62
Moisture content, mol water per mol dry gas	0.19	0.56

*At 60°F, 30-in. Hg, dry.

contact between the oil shale and electrode always is of marked importance.

Electrogasifying appears to be a suitable comprehensive method for the treatment of oil shale *in situ*. It involves electrical loosening of the formation to develop reaction channels; the conversion then can be continued by combustion of fixed carbon. The several temperature levels formed in the fuel bed in thick and/or intercalated oil shale deposits can be used as heating elements for continuous carbonizing of surrounding oil-bearing material. Electrical carbonizing should be used as long as economically feasible, before the air is introduced and the fixed carbon burned. The economic limit varies as a function of the oil content in the oil shale and the cost of available electricity.

Additional field experimentation with electrocarbonization of buried oil shale must be done and extensive research accomplished before it will be possible to apply the process on an industrial scale. Preliminary laboratory tests[14] made on a sample of brown coal indicate that results may be achieved which are broadly similar to those obtained by treating oil shale.

Application of the general process of electrocarbonization for stimulating and increasing the recovery of crude oil from stripper fields, depleted oil pools, and tar sand deposits such as at Athabasca, Canada, was recognized by the authors when research on underground gasification of coal was begun in September 1947. The procedure, which may be classed as a *tertiary recovery* method for securing oil by subsurface electrical heating, is of such unique character as a phase of electrocarbonization that it has been named the *electrotherm* process. Since even the most specialized methods of secondary recovery as applied in oil reservoir rocks commonly fail to secure more than 60 pct of the potential oil, this electrotherm process, which can extend this production ratio, is important and merits distinction.

The first subsurface electrical treatment of oil sand was performed by Sarapuu[14] on material from Elsas Lothringen, where the famous French oil sand mines are located. This laboratory experimentation was done by extensive heating, and for practical purposes the oil was distilled and procured in gaseous form. This was possible because of the low electric resistance of residual fixed carbon in the oil sand. Further laboratory investigations were made in the mining engineering department at Missouri School of Mines in the period from 1947 to 1951 when several researchers[15-17] studied the behavior of oil sands subjected to electrical heating and combustion. This laboratory research created marked interest, and, apparently as a result, C. M. Davis[18] and Trevor W. W. Hill[19] subsequently undertook to carry on their own research elsewhere.

The results described by Davis and Hill appear to follow the general pattern of those which attend the electrotherm process. However, the authors of this paper cannot agree with their re-theorizing of the process. Their attempts to explain the phenomena of electrocarbonization as being primarily that of dielectric breakdown are not in accordance with the proved scientific facts. The new terms they propose, such as *pyrovolatilization, electrovolatilization,* and *dielectric heating,* actually are synonyms of *electrolinking, electrogasifying,* and *electrocarbonizing.* In general terms, it appears that the breakdown they ascribe to dielectric heating is one resulting from mechanical failure of insulators in the system. Electrocarbonizing is not a localized phenomenon, and it may extend through the raw fuel deposit for several hundred feet. It is a process whereby the chemical nature of the raw material is changed, and thus the original high resistivity is reduced. Electrocarbonizing is a resistance heating and cannot be explained as dielectric heating.

Although adaptation of the electrotherm process is feasible, there are several aspects that make the process somewhat different from that of electrocarbonization which applies to the treatment of coal *in situ*. For example, the primary aim with petroleum-bearing deposits is to secure the production of liquid substances rather than gaseous materials. Also, geological environments and modes of occurrences of oil-bearing deposits commonly are much more diversified than those of coal, and this results in a need for applying special techniques to individual oil pools. Tertiary recovery methods of oil field exploitation are predicated on the use of the thermal energy to provide a means of driving hydrocarbon fluids from the reservoir rock. That is, if enough energy in the form of heat can be injected into the deposit, it will provide necessary expulsive forces to drive some of the fluid from the reservoir.

The advantages of the electrotherm process in oil recovery are the complete controllability of the operation and the opportunity to concentrate a range of heat in the reservoir rock at a given well location. The application of heat will produce oil progressively, and this can be done most effectively in two or three stages. Each successive rise of temperature should begin when further recovery is no longer possible at the previous stage. For example, low-temperature heating will induce fluidity and will dissipate congealed hydrocarbons that often inhibit the movement of liquids through the rock. Surface tension of oil and the interfacial tension between oil and water will be reduced. Thus it is apparent that low-range heat may stimulate recovery from stripper wells or from otherwise depleted sands.

When the high temperature of electrotherm treatment is required, the electric current is passed directly through the oil reservoir and the extent of heat penetration is greater than that given by other methods. High temperatures fracture the oil-bearing rocks; viscosity is lowered; and the evaporation of fluids supplies additional energy which in turn, by disturbing the equilibrium in the reservoir, will support and increase the flow.

It is possible that the most economical industrial process ultimately may be found to involve contemporary electrogasification and distillation of the oil. That is, it may be desired commercially to accomplish stages of tertiary recovery by utilizing temperatures of various ranges. The heating should be done in a continuous cycle until complete recovery of the hydrocarbon potential eventually is obtained by electrogasification. The electrodes to be used must be designed, of course, to withstand the temperatures that will be required to accomplish the whole cycle.

To summarize, it may be noted again that the geological environments and modes of occurrence of oil deposits are so numerous and diversified that it is impossible to prescribe a treatment that will apply to all cases. However, electrocarbonization as a whole, or as it is here designated, the *electrotherm process,* is a very flexible method and can be used as follows:

1—To stimulate recovery of oil from stripper fields or depleted pools by melting and removing the con-

gealed hydrocarbon constituents in the sands that otherwise restrict and inhibit the flow of oil to well.

2—To stimulate the recovery from depleted fields and stripper areas by means of electrical heat to induce the development of linkages and channels in the reservoir rock.

3—To stimulate the flow of oil from highly viscous oil sand deposits by the gasification, in part at least, of residual oil.

4—To secure gaseous hydrocarbon compounds from depleted fields after all other production methods have been applied and exhausted. This process entails complete gasification *in situ* by use of electrical heat.

References

[1] P. Averitt, L. R. Berryhill, and D. A. Taylor: *Coal Resources of the United States* (A progress report, Oct. 1, 1953) USGS *Circ. 293*, p. 40.

[2] USGS *Circ. 94* (1950).

[3] J. C. Sproule: The McMurray Formation in Its Relation to Oil Occurrence. *Proc.* Athabasca Oil Sands Conference, Alberta, Canada (September 1951) p. 6.

[4] *Synthetic Liquid Fuels, Annual Report of the Secretary of the Interior for 1952*, Part II. *Oil from Oil Shale*, U. S. Bur. Mines *R. I. 4943* (January 1953) p. 1.

[5] A. J. Kraemer: *Oil Shale in Brazil.* U. S. Bur. Mines *R. I. 4644* (February 1950) p. 16.

[6] *Proved Reserves of Crude Oil, Natural Gas Liquids and Natural Gas*, 7, p. 9. Published by API and AGA, Dec. 31, 1952.

[7] *World Crude Reserves: World Oil-International Operations Issue* (Aug. 15, 1952) p. 77.

[8] J. D. Forrester and Erich Sarapuu: The Process of Underground Electrocarbonization, *Bulletin*, Missouri School of Mines and Metallurgy, Technical Series, No. 78 (January 1952) pp. 10, 21.

[9] J. D. Forrester: Electrocarbonization of Coal and Related Hydrocarbons, Part I. *Mechanization* (April 1953); Part II, *Mechanization*, June 1953.

[10] E. Sarapuu and P. C. Cross: *The Underground Electrocarbonization of Coal.* American Institute of Electrical Engineers, Kansas City, Mo., Nov. 4, 1953.

[11] Westinghouse Electric Corp.: *Summary Report of Blast Furnace Gas Combustion Investigation*, November 1953.

[12] M. F. Fies and J. L. Elder: Underground Gasification of Coal at Gorgas, Alabama. ASME Paper No. 53-A-137.

[13] E. Sarapuu: *The Investigation to Gasify Mineral Fuels in Situ-Electrocarbonization.* Thesis, Mining Academy, Freiberg, Germany, 1945.

[14] E. Sarapuu: *Subterraenean Carbonization of Oil Shale, etc.* Technical Oil Mission Reel 242, Item 37, Library of Congress, Washington, D. C., 1947.

[15] H. R. Coffer: *Thermal Recovery of Petroleum by Electric Heating.* Thesis, Missouri School of Mines and Metallurgy.

[16] C. M. Davis: *Some Preliminary Investigation Toward the Use of Electrical Energy for Producing Petroleum.* Mining Industries Experimentation Station, Bull. No. 60, The Pennsylvania State College.

[17] C. M. Davis: The Advent of Electrovolatilization. *Producer's Monthly*, May 1951.

[18] C. M. Davis: Electrovolatilization of Oil in Situ. *Proc.*, Athabasca Oil Sands Conference (September 1951).

[19] T. W. W. Hill: Electro-Thermal Recovery of Petroleum. *Producer's Monthly* (September 1952).

Depth Determinations by Electrical Resistivity

by Harold M. Mooney

RESISTIVITY measurements for determining depth to bedrock, water table, and other geologic discontinuities have had only limited success. Many of the difficulties can be attributed to complex geology and to basic limitations of the method; current flow through a continuous medium is a diffusion phenomenon, emphasizing irregularities near the source and smoothing out those at a distance. Some of the failures, however, are due to careless field work and incompetent interpretation. A forthcoming publication[1] may help to reduce difficulties of this sort.

The writer here attempts to bring order into the multiplicity of interpretation methods and, to a limited extent, to evaluate them. For all methods, the field data to be interpreted consist of apparent resistivity values plotted against electrode separation, Fig. 1. The interpretation should give depths to one or more geologic horizons. Direct currents and the Wenner four-electrode configuration will be assumed, since these are widely used, although the interpretation problems remain essentially the same for other electrode configurations. Alternating currents higher than, say, 20 cycles per second introduce complications beyond the scope of this paper.

H. M. MOONEY is Assistant Professor of Geophysics, University of Minnesota, Minneapolis.

Discussion on this paper, TP 3890I, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript, March 3, 1954. New York Meeting, February 1954.

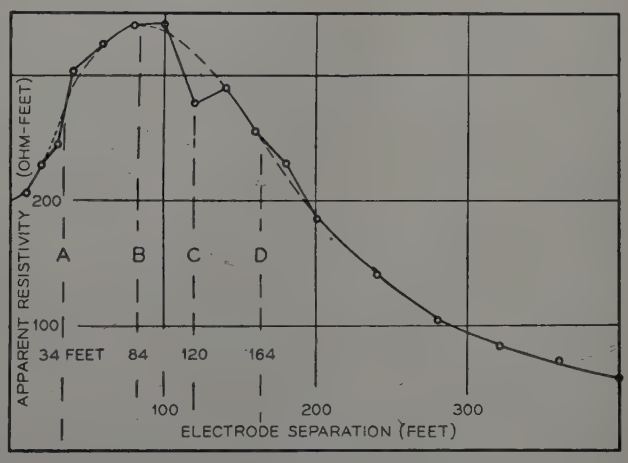


Fig. 1—Typical resistivity field data.

Three approaches to the interpretation problem have been used. These may be called empirical, theoretical curve matching, and direct. Many techniques have been proposed, but each can be reduced to one of these approaches and to a single geologic idealization. This idealization rests on the following assumption: the earth in the limited volume of interest can be approximated by a horizontal sequence of layers, each homogeneous and isotropic in its electrical properties. This seemingly restrictive assumption has proved to have practical value, although future work should aim at removing some

of the restrictions. An attempt in this direction forms the subject of a separate paper.[2]

The field curve normally shows maxima, minima, points of inflection, and breaks (sudden changes in slope). Empirical methods select some such characteristic and equate the corresponding electrode separation to the depth to a discontinuity or to a fraction thereof.

The first and most widely used empirical method was proposed by Gish and Rooney.[3] They equate the depth to the electrode separation at which a maximum or minimum occurs. (In Fig. 1, depth B = 84 ft). Lugeon and Schlumberger[4] prefer three-fourths of this electrode separation. (In Fig. 1, depth B = 63 ft). The method can give order-of-magnitude results over a restricted area, provided the multiplying factor is established by drill control.

A common perversion of this technique uses breaks in the curve rather than maxima or minima. (In Fig. 1, depth C = 120 ft). Such breaks can be due only to lateral changes, electrolytic phenomena, or instrumental troubles. They can not be due to the vertical changes of interest.

Lancaster-Jones[5] takes a point of inflection as the observable characteristic. He equates the depth to two-thirds of the corresponding electrode separation. (In Fig. 1, depths A and D = 23 and 109 ft). Palmer and Hough[6] point out that this applies to only a single discontinuity and a particular ratio of resistivities. They extend the technique to other cases, but recognize that it has limited application.

Another group of empirical methods modifies the field curve before it is interpreted. A suggestion of Moore[7] has aroused some controversy. Moore's cumulative resistivity method requires data taken at equal increments of electrode separation. For each separation he plots as the measured quantity the sum of that reading plus all preceding apparent resistivity readings. He draws straight lines through the resulting points, and equates electrode separations at the line intersections to depths to discontinuities, Fig. 2.

In effect, Moore plots the integral of the apparent resistivity curve. This produces a smoothing of the data, which can be justified for some purpose, and a greatly reduced vertical scale. The interpretation

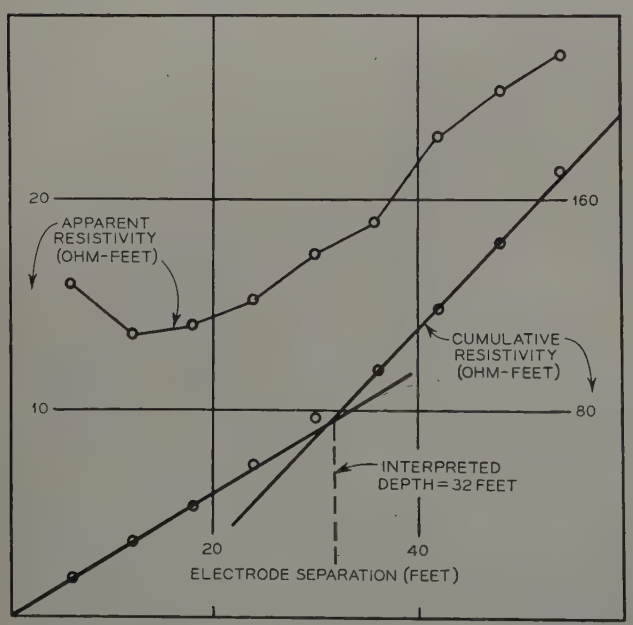


Fig. 2—An example of resistivity interpretation by Moore's method.

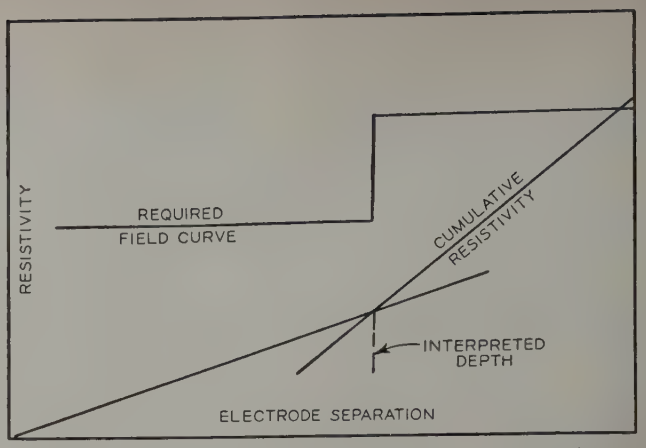


Fig. 3—One implication of Moore's method of interpretation, in an ideal situation.

based on straight lines makes no theoretical sense, however, as Muskat[8] has shown and as Moore readily admits. The simplest integral curve, that of two intersecting straight lines, would be produced by an apparent resistivity curve consisting of two horizontal segments (constant apparent resistivity), abruptly offset, Fig. 3. No such curve has been observed or predicted. The Moore method probably reduces to a modified Gish-Rooney technique, with possibly some advantage through smoothing. Ruedy[9] has proposed a theoretical justification for Moore's method, but it is hard to see how his conclusions follow from his data.

A second method which modifies the field data has been suggested by Barnes.[10] The analysis is obscure and unnecessarily complicated, but Rogers[11] has shown that he plots an approximation to the reciprocal slope of the conductivity depth curve. The interpretation reverts to the Gish-Rooney method.

Other methods have been proposed which require graphical differentiation of the field curve. This gives sharper indications but emphasizes accidental irregularities. Potential drop ratio methods approximate this scheme; data can be interpreted by curve matching or empirically. For empirical interpretation, depths are set equal to those electrode spacings where the field curve passes through a standard value. This is no better and no worse than the Gish-Rooney method.

Given an assumed sequence of layers, with specified thicknesses and electrical properties, a theoretical resistivity curve can be calculated. A variety of such assumptions will yield a family of curves. If the field curve can be shown to match some one of this family, then the actual geology presumably approximates the idealized conditions used in calculating the curve. The quality of the match provides a rough confidence estimate.

The field curve and the type curves can be plotted on either a linear (cartesian) or a logarithmic scale. According to Heiland,[12] the Schlumberger organization at one time used direct curve matching on a linear scale. A more widely used linear method was proposed by Tagg.[13] Starting from calculated curves for a single horizontal discontinuity, he describes a trial and error process which yields depth and resistivity values. The method requires an independent determination of surface resistivity. Later proposals have eliminated this requirement by using apparent resistivity ratios. A later paper by Tagg[14] and papers by Rosenzweig[15] and Longacre[16] describe methods of this sort. All of these require extensive analysis to interpret each field curve.

The logarithmic scale has such obvious advantages that linear methods such as Tagg's should be abandoned. 1—The shape of the curve becomes independent of the units of resistivity or electrode separation. A single logarithmic curve can replace a large number of linear curves, since the latter must be replotted for each change in scale of the problem. Further, the interpreter can soon develop an intuitive feel for curve trends. 2—The problem of determining near-surface resistivity becomes easier, since the entire first portion of the curve can be used.

Specific suggestions for interpretation by logarithmic curve matching are given by Roman,[17] Wetzel and McMurry,[18] and particularly by Spicer.[1] Wetzel and McMurry also present tables for plotting two and three-layer curves. The same graph paper must be used for field and calculated curves; two-cycle log paper such as Dietzgen 340D-L23 or Keuffel and Esser 359-112L is recommended. Two, three, and four-layer curves will be made available by Mooney and Wetzel.[19]

Fig. 4 shows the field data of Fig. 1 on a logarithmic scale. Fig. 5 shows a group of three-layer theoretical curves. When curves are superposed, the best match occurs for the heavy curve in Fig. 5, which was calculated for resistivity ratio 1:3:1/10 and depth ratio 1:4. The unit axes cross the field curve at 97 ft (this gives depth to the second interface) and 192 ohm ft (surface resistivity). The complete interpretation gives two discontinuities, at depths of 24 and 97 ft, separating media with true resistivities 192, 576, and 19 ohm ft.

A field curve can rarely be made to match completely a calculated curve, because of the limited number of calculated curves available. Interpolation between them becomes necessary. Fortunately the properties of a logarithmic plot make this feasible. An experienced interpreter has a feel for the behavior of resistivity curves under various conditions; one important use for calculated curves, in fact, is to provide a sound basis for developing such a feel quickly.

Many field curves clearly represent four layers or more. A second problem which arises in curve matching is to interpret these by two and three-layer curves, until four-layer curves become available. The basis for the method was suggested by Hummel.[20] He proposes to reduce two surface layers to a single equivalent layer, using an analogy with parallel resistances in an electrical circuit. He notes that the approximation applies only for electrode

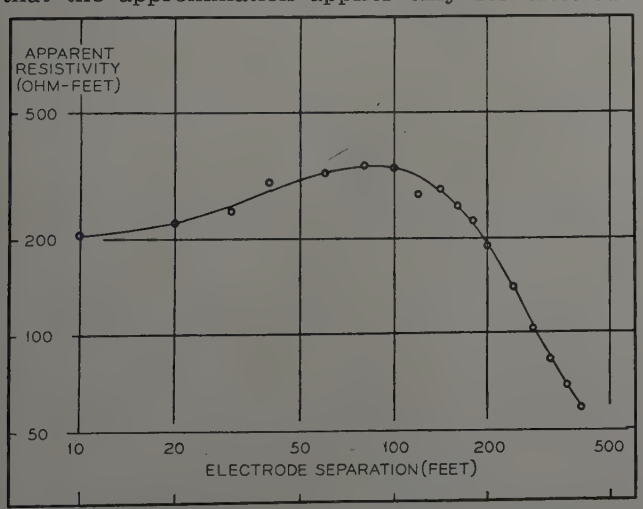


Fig. 4—Resistivity data of Fig. 1, replotted on a double logarithmic scale.

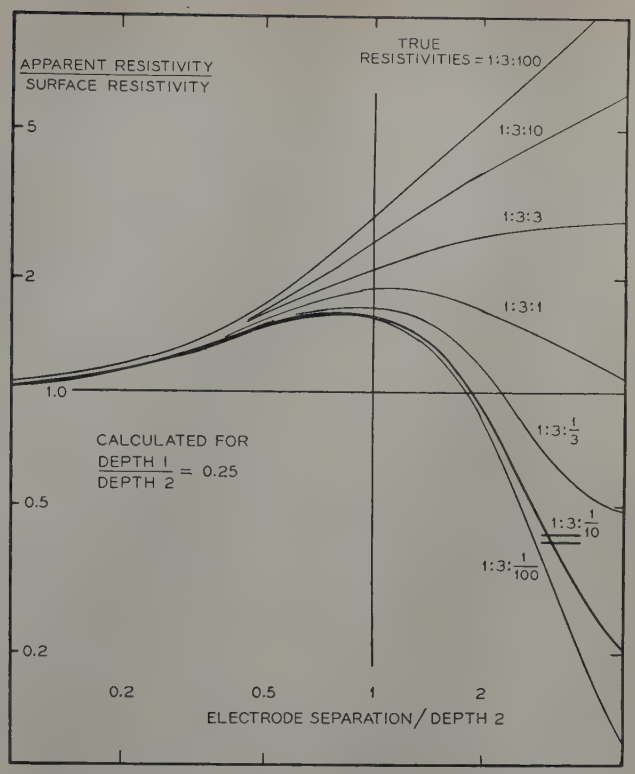


Fig. 5—A group of theoretical curves, calculated for one depth ratio and several resistivity combinations.

spacing that is large compared with the thicknesses of the two surface layers. Pirson[21] combines this with Tagg's method and a method of successive approximations. The fact that Hummel's rule can lead to serious error has been noted by Geneslay and Rouget.[22] Ebert[23] derives four such rules for the three-layer case, based on possible resistivity ratios in the layers; one of these reduces to Hummel's rule.

The use of Hummel's rule requires a partial match. For example, the first part of a three-layer curve can sometimes be fitted to one two-layer calculated curve, and the last part to another. Wetzel and McMurry[18] conclude that this may give fair accuracy when the three-layer curve has a minimum, but usually fails when it has a maximum. They point out further that two-layer data do not always represent a limiting case for a particular set of three-layer curves, but that the curves may in fact cross. Watson and Johnson[24] conclude that the second layer should be appreciably thicker than the surface layer to apply successfully a partial match.

Compared with empirical methods, curve matching has the following advantages: 1—It will usually give no answer rather than an incorrect one, in cases where the true geology deviates so widely from the assumptions that resistivity methods are inapplicable. This is an outstanding weakness of Moore's method, for example, which will almost always give some sort of result. 2—The entire resistivity curve, rather than localized portions of it, is used to obtain the geologic data. 3—The quality of a match provides an estimate for the reliability of an interpretation. 4—The method is theoretically exact within the assumptions, and the assumptions are clearly stated. Extensions of the method to less restrictive assumptions can be made with confidence. 5—For the two-layer case (the usual limit for empirical methods), curve matching is as simple as empirical technique and also faster, considerably more so than the Moore method.

The utility of curve matching should not be exaggerated, however: 1—Extrapolation between curves is usually necessary, which requires judgment based on experience. 2—Many field curves give no match at all. This may be due to failure of the field situation to satisfy the assumptions, or to carelessness in taking the data. Either lateral or vertical variations may cause the field situation to differ from the assumption; lateral variations can be detected by offsetting or rotating the spread.

Perret[25] and Petsch[26] give field comparisons of the various interpretation methods.

Direct Methods

Two interpretation methods have been proposed which work directly from the field data to geologic layering. No calculated curves are required as in curve matching, yet the methods have more theoretical basis than the empirical techniques.

Evjen[27] has proposed a weighting method which deals with image densities rather than with depths to discontinuities. The treatment can be kept rigorous to a later stage in the interpretation, yet it ultimately requires approximations which may or may not be satisfactory. Roman[28] mentions unpublished work on weighting.

Slichter[29] suggested a direct process based on an integral transform, and Pekeris[30] described the steps necessary to apply this to a layered earth. In calculation of potentials about a point source in a layered medium, this seems to bear the same relation to Evjen's proposal that the integral representation does to the image representation.

The steps required by Pekeris are: 1—Measure the potential distribution about a point electrode, by some necessarily indirect process. 2—Perform a numerical integration, involving the potential as one term, over distance from the electrode. 3—Interpret the results graphically in terms of resistivities and of depths to discontinuities.

The entire method has been investigated in some detail by Madden, Rogers, and Mooney.[31] A satisfactory field procedure was developed to yield potentials about a point electrode. Tables suitable for use on an electronic computer were calculated, based on a polynomial approximation, to carry out the numerical integration. The approximation of an infinite integral by a finite set of measurements proved particularly troublesome. The method was applied to nine sets of calculated potential data. Two alternatives to Pekeris' method were developed.

They concluded that: 1—The Pekeris method involves a restrictive assumption, namely that each layer must be thicker than the one above it. 2—For a multi-layer problem, accuracy for successive layers drops off sharply. This arises in part because the solution for each layer depends on all previous solutions. 3—The results for even the first layer are not consistently satisfactory. Of the nine cases mentioned above (two three-layer and seven four-layer), seven yielded a good solution for the top layer, but only two for the next layer. 4—The method is laborious, complicated, and hard to associate with the geologic problem.

These tentative results should not be taken as conclusive. Other groups now working on the problem may develop a fresh approach with more success.

References

[1] H. C. Spicer: Handbook for Resistivity Exploration Using Gish-Rooney Equipment. U. S. Geol. Survey *Bull.* In preparation.

[2] H. M. Mooney: Effect of a Variable Surface Layer on Electrical Resistivity. *Trans.* AIME. In press.

[3] O. H. Gish and W. J. Rooney: Measurement of Resistivity of Large Masses of Undisturbed Earth. Territorial Magnesium and Atmospheric Electricity (1925) **30**, p. 261.

[4] M. Lugeon and C. Schlumberger: Electrical Study of Dam Foundations. *Mining Magazine* (1933) **48**, p. 340.

[5] E. Lancaster-Jones: The Earth-Resistivity Method of Geophysical Prospecting. *Mining Magazine* (1930) **42**, p. 352; (1930) **43**, p. 19.

[6] L. S. Palmer and J. M. Hough: Geoelectrical Resistivity Measurements. *Mining Magazine* (1953) **88**, p. 16.

[7] R. W. Moore: An Empirical Method of Interpretation of Earth Resistivity Measurements. *Trans.* AIME (1945) **164**, p. 197.

[8] M. Muskat: Interpretation of Earth Resistivity Measurements. *Trans.* AIME (1945) **164**, p. 224.

[9] R. Ruedy: The Use of Cumulative Resistance in Earth Resistivity Surveys. *Canadian Journal of Research* (1945) **23A**, p. 57.

[10] H. E. Barnes: Soil Investigation Employing a New Method of Layer Value Determination. Bulletin Michigan Highway Department (1951).

[11] G. R. Rogers: Personal communication, June 30, 1953.

[12] C. A. Heiland: *Geophysical Exploration.* Prentice-Hall Inc., New York, 1940.

[13] G. F. Tagg: Interpretation of Earth Resistivity Curves. *Trans.* AIME (1934) **110**, p. 135.

[14] G. F. Tagg: Interpretation of Earth Resistivity Curves. *Trans.* AIME (1940) **138**, p. 399.

[15] I. E. Rosenzweig: A New Method of Depth Determination. *Trans.* AIME (1940) **138**, p. 408.

[16] W. A. Longacre: A Study of the Problem of Depth Determination by Resistivity Measurements. *Trans.* AIME (1945) **164**, p. 179.

[17] I. Roman: Superposition in the Interpretation of Two-Layer Earth Resistivity Curves. U. S. Geol. Survey *Bull. 927-A* (1941) 18 pp.

[18] W. W. Wetzel and H. V. McMurry: A Set of Curves to Assist in the Interpretation of the Three-Layer Resistivity Problem. *Geophysics* (1937) **2**, p. 7.

[19] H. M. Mooney and W. W. Wetzel: Potentials about a Point Electrode and Apparent Resistivity for a Four-Layered Earth. Scheduled for 1955 publication, University of Minnesota Press.

[20] J. N. Hummel: Apparent Resistivity. *Zeits. Geoph.* (1929) **5**, pp. 89, 228. In German.

[21] S. J. Pirson: Interpretation of Three-Layer Resistivity Curves. *Trans.* AIME (1934) **110**, p. 148.

[22] R. Geneslay and F. Rouget. *On Electrical Anisotropy,* p. 723. 2me Cong. Mond. Petr., Paris, 1937. In French.

[23] A. Ebert: Basis of Electrical Depth Determinations. *Beitr. Ang. Geoph.* (1942) **10**, p. 1. In German.

[24] R. J. Watson and J. F. Johnson: On the Extension of Two-Layer Methods of Interpretation of Earth Resistivity Data to Three or More Layers. *Geophysics* (1938) **3**, p. 7.

[25] W. R. Perret: Electrical Resistivity Exploration. Waterways Experiment Station, Vicksburg, Miss. *Bull. 33* (1949) 48 pp.

[26] B. C. Petsch: A Geophysical Study of the Milbank Granite Area. South Dakota Geol. Survey *R.I. 60* (1948) 21 pp.

[27] H. M. Evjen: Depth Factors and Resolving Power of Electrical Measurements. *Geophysics* (1938) **3**, p. 79.

[28] I. Roman: Resistivity Reconnaissance. American Society of Testing Materials Special Tech. Pub. 122 (1952) p. 171.

[29] L. B. Slichter: The Interpretation of the Resistivity Prospecting Method for Horizontal Structures. *Physics* (1933) **4**, p. 307.

[30] C. L. Pekeris: Direct Method of Interpretation in Resistivity Prospecting. *Geophysics* (1940) **5**, p. 31.

[31] T. R. Madden, G. R. Rogers, and H. M. Mooney: Unpublished manuscript, 1952.

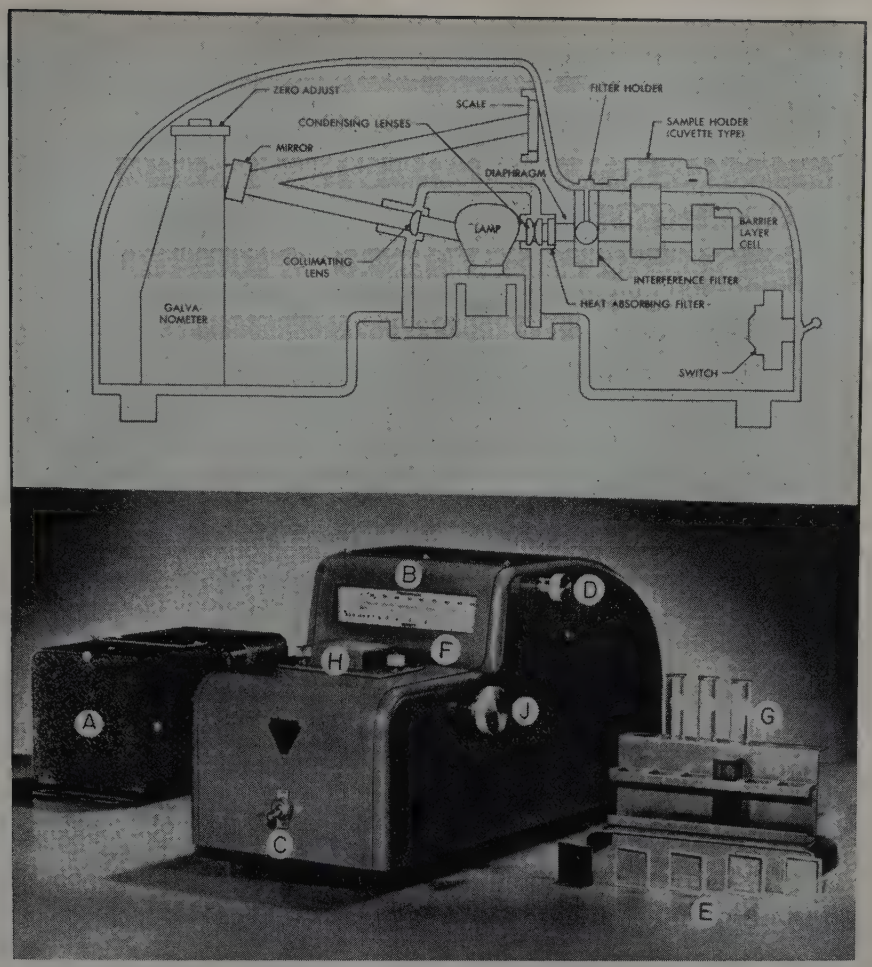


Fig. 1—The Bausch & Lomb monochromatic colorimeter: *A*—Transformer. *B*—Transmission scale. *C*—Toggle switch. *D*—Control knob. *E*—Interference filter. *F*—Filter channel. *G*—Test tube. *H*—Sample space. *J*—Adjustment knob.

Rapid Estimation of Mill Product Purity By Transparency Measurement

by S. C. Sun, H. M. Fisher, and R. E. Snow

RAPID colorimetric estimation of the amount of transparent minerals has been applied successfully to instream mill products at the cryolite flotation plant of the Pennsylvania Salt Mfg. Co., Natrona, Pa. Apparatus used was a Bausch & Lomb monochromatic colorimeter. The optical assembly of the colorimeter, shown in Fig. 1, passes light from a 6-v, 32-cp, prefocus incandescent lamp through two condensing lenses, a heat-absorbing filter, an aperture-control diaphragm, an interference filter, and a sample container. The light finally strikes a barrier-layer photocell and the output current of the photocell is measured by a galvanometer. The engraved index line of the collective lens, located between the lamp and the galvanometer, is imaged upon the transmission scale of the colorimeter by the galvanometer mirror. Reflected light from the galvanometer mirror, the position of which is determined by the output current of the photocell, enables reading of percent transmission throughout the mineral sample being tested.

The colorimeter, Fig. 1, was standardized as follows: 1—The colorimeter was connected to a Sola constant voltage transformer, *A*, of 6-v output operated from a 110-v ac supply. 2—Turning the toggle switch, *C*, illuminated the transmission scale, *B*, and the control knob, *D*, was adjusted with a blank in the optical system until the hairline on the scale read zero. 3—A No. 430 interference filter, *E*, was inserted into the filter channel, *F*. 4—A test tube, *G*, containing 7 cc of distilled water was pressed into the sample space, *H*, and the fine adjustment knob,

S. C. SUN, Member AIME, is Associate Professor of Mineral Preparation, The Pennsylvania State University, State College, Pa. H. M. FISHER is Development Engineer, Natrona Plant, Pennsylvania Salt Mfg. Co., Natrona, Pa. R. E. SNOW is a Graduate Student, The Pennsylvania State University.

Discussion on this paper, TP 3819B, may be sent (2 copies) to AIME before Oct. 31, 1954. Manuscript, Nov. 9, 1953. New York Meeting, February 1954.

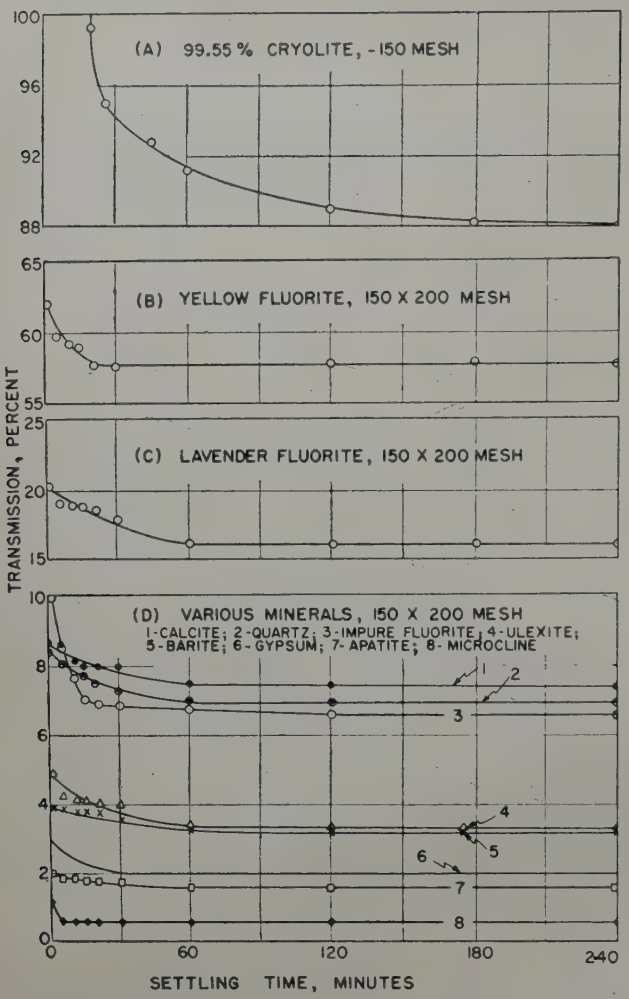

(A) 99.55% CRYOLITE, -150 MESH
(B) YELLOW FLUORITE, 150 X 200 MESH
(C) LAVENDER FLUORITE, 150 X 200 MESH
(D) VARIOUS MINERALS, 150 X 200 MESH
1-CALCITE; 2-QUARTZ; 3-IMPURE FLUORITE; 4-ULEXITE; 5-BARITE; 6-GYPSUM; 7-APATITE; 8-MICROCLINE
TRANSMISSION, PERCENT
SETTLING TIME, MINUTES

Fig. 2—Transparency of various mineral sediments after different settling times.

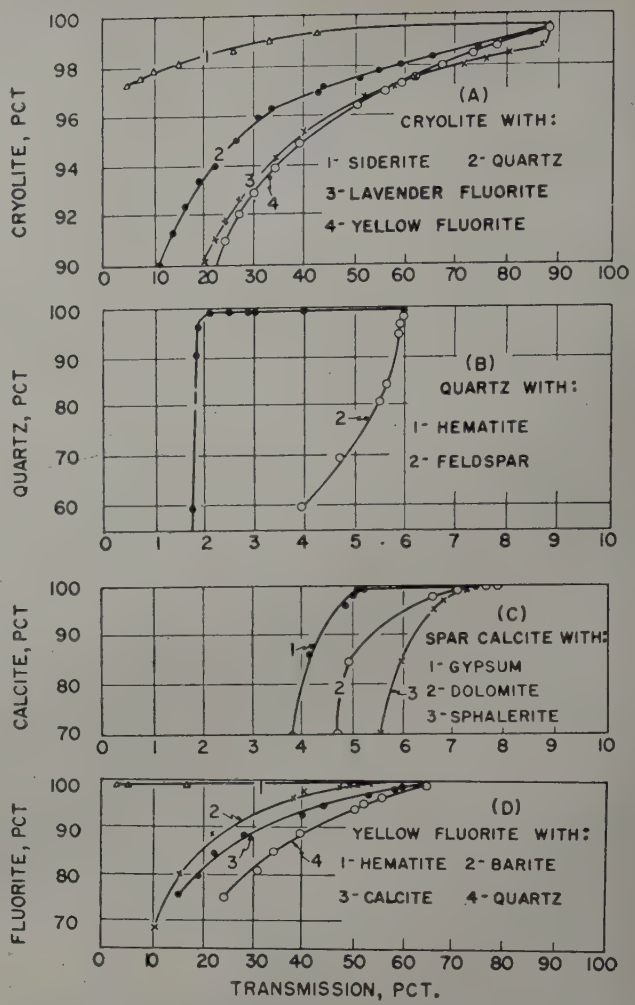


Fig. 3—Effect of mineralogical composition on transparency of mineral sediments. All minerals used —150 mesh.

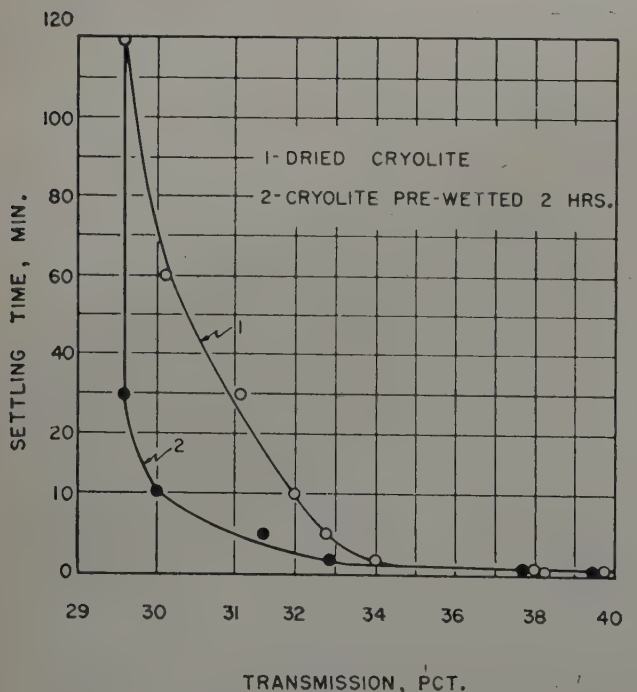


Fig. 4—The effect of settling time on transparency of —65 mesh cryolite sediments.

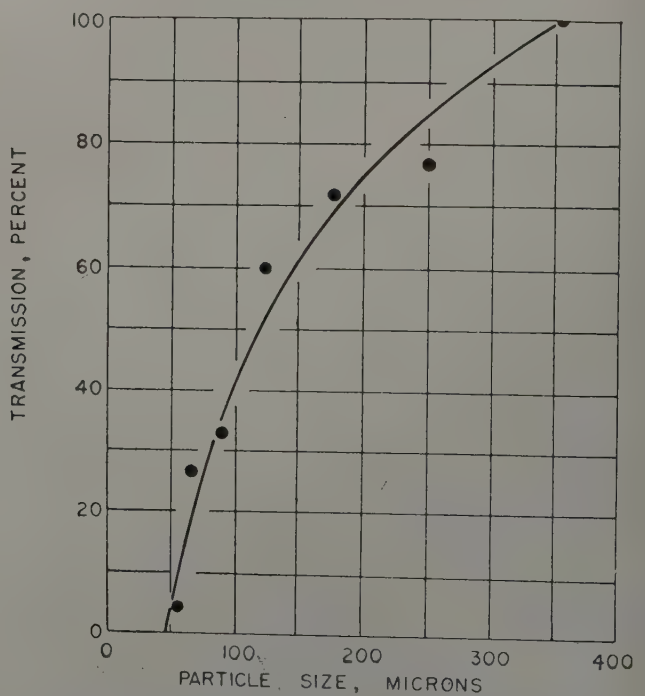


Fig. 5—The effect of particle size on the transparency of cryolite sediments.

J, turned until the hairline was set at a predetermined point of the transmission scale. 5—The test tube and then the filter were removed from the colorimeter.

The distilled water standard setting used for the highly transparent cryolite samples was arbitrarily chosen as 10, and for the less transparent minerals 100. A setting of 100 admitted too much light for measuring the cryolite samples, resulting in off-scale deflection.

The experimental procedure for measuring transparency of mineral sediments was carried out as follows: 1—A predetermined amount of sized mineral sample, usually 4 g or more, was weighed out and placed in a test tube of 1.2 cm OD to form a mineral column not less than 2.1 cm high. 2—Four cubic cm of distilled water were added to the test tube and shaken for 1 min; 3 cc. of distilled water were then added and shaken for 2 min. 3—The test tube containing this mineral pulp was inserted into the sample space of the standardized colorimeter, and the transmission scale reading was recorded at various time intervals until the reading became constant. Unless otherwise stated data presented here were taken after 4 hr settling.

Figs. 2-5 indicate that while the light source and the mineral container are kept constant, the transparency of mineral sediments is chiefly governed by: 1—mineralogical composition, 2—settling time, and 3—particle size of the sediment. Concerning point 1, Fig. 2 shows that the transparencies of individual mineral sediments differ appreciably; generally speaking, transparencies decrease in the order of cryolite, fluorite, calcite, quartz, impure fluorite, ulexite, gypsum, and microcline. This is because the absorption coefficient and/or the refractive index of these minerals increase in the same order. The absorption and reflection of light by a mineral increase respectively with an increase of the absorption coefficient and the refractive index[1] of the mineral. When light is sent through a column of mineral sediment, the incident light is partly absorbed and partly reflected and only the remainder is transmitted and measured. Fig. 3 shows that the transparency of a considerably transparent mineral sediment is gradually lowered when it is blended with increasing amounts of a less transparent mineral. This phenomenon becomes even more pronounced when the added mineral particles are opaque. Thus, under restrictive conditions, the purity of a transparent mineral can be estimated by means of transparency measurement.

Concerning point 2, Fig. 2 shows that the transparency of a mineral sediment decreases with settling time until a constant transmission is attained. A 2-hr settling time was sufficient to establish a constant transmission for all minerals tested, except that 4 hr were necessary for the —150 mesh cryolite. In contrast, Fig. 4 shows that the necessary settling time of —65 mesh cryolite is 2 hr. Furthermore, when the same cryolite sample is pre-wetted in distilled water and allowed to settle a second time, the necessary settling time is reduced to only ½ hr. The disadvantage of the long settling time required was eliminated by centrifuging the mineral pulp for 10 min at 2850 rpm and allowing the compacted sediment to stand for 5 min before transparency measurements were taken. The relation between transmission and settling time can be explained by the fact that the interstices of mineral sediment decrease gradually with an increase of set-

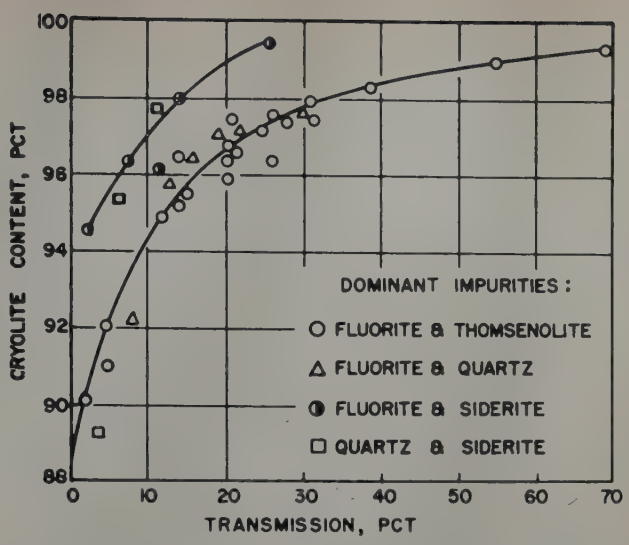


Fig. 6—Effect of the type of dominant impurities on the transparency of cryolite flotation concentrates. Note that the cryolite samples used were prepared from different ores and were all —150 mesh.

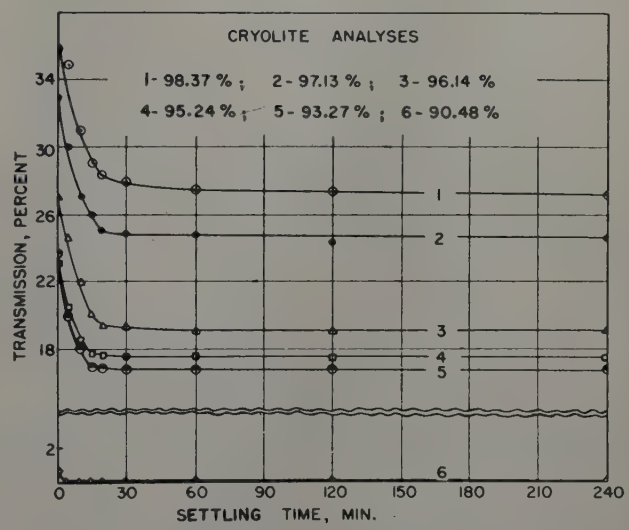


Fig. 7—Estimation of the grade of cryolite flotation concentrates. Note that all cryolite samples were prepared from the same ore and were —150 mesh.

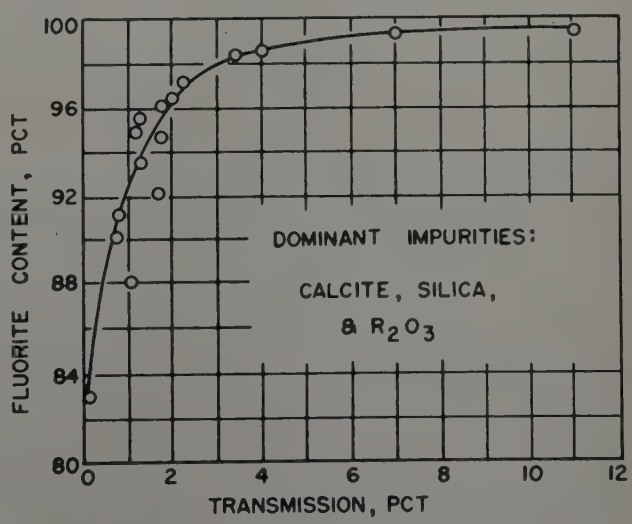


Fig. 8—Estimation of grade of fluorite flotation concentrates. Note that the fluorite samples were furnished by two different mills and were all —100 mesh.

tling time and eventually become constant. These interstitial voids filled with distilled water are highly transparent. The fact that some minerals require more settling time than others for establishing constant transmission is attributed to their difference in density and wettability.

With regard to point 3, Fig. 5 shows that the transparency of a mineral sediment, within certain limits, increases with an increase of the particle size of the mineral. This is because the interstices between the particles of a coarse sediment are larger than those of a fine sediment.

The data of Figs. 6-8 indicate that under restrictive conditions a monochromatic colorimeter can be employed for a quick estimation of the content of any transparent mineral in mill products, resulting in a better control of plant operation. Fig. 6 shows that siderite is more effective than other impurities tested in reducing the transparency of cryolite sediments. This implies that the transparency method is applicable only when the mineralogical composition of the dominant impurities of cryolite concentrates is pre-determined. Fig. 7 shows that when the impurities of cryolite samples are dominated by the same gangue minerals, the difference in cryolite content of the samples is revealed by the transparency method. It should be noted that the samples of Fig. 6 were prepared from four different types of cryolite ore, whereas all samples of Fig. 7 were derived from one ore.

Fig. 8 shows that under suitable conditions it is also possible to estimate the grade of fluorite concentrates by the transparency method. Figs. 6 and 8 indicate also that as now designed the colorimeter used in this investigation cannot give accurate measurements for samples containing less than 90 pct cryolite or fluorite because of their low transmission. However, this is not a serious drawback, as the acceptable grades of the flotation products of cryolite and acid-grade fluorite are respectively higher than 96.5 and 92 pct and are thus far above the lower limit of the colorimeter. Furthermore, this limitation can be partly eliminated by increasing the light intensity and the transmission scale of the colorimeter, reducing the diameter of the sample container, or both.

Summary

1—A transparency method is described for estimating the grade of cryolite and fluorite concentrates under restrictive conditions.

2—The colorimeter used in this investigation has been found useful in the estimation of the grade of concentrate of highly transparent minerals, but a colorimeter possessing greater light sensitivity would be necessary for estimation of grade of the concentrate of the less transparent minerals.

Acknowledgments

The writers wish to thank D. C. Spees of the Fluorspar Division, Minerva Oil Co., Eldorado, Ill., for providing the fluorite samples. They also wish to thank L. Goldenhar, J. F. Gall, D. R. Mitchell, A. E. Clement, and H. B. Charmbury for their active interest in this work.

Reference

[1] E. Hausmann and E. P. Slack: *Physics*, 2nd ed., p. 618. D. Van Nostrand Co., Inc., 1939.

Substituted Starches in Amine Flotation of Iron Ore

by C. S. Chang

Replacement of active groups in corn starch, as shown by results of the starch derivatives tested, impairs rather than improves the value of corn starch as a selective iron oxide depressant. Increased ability to depress iron oxides is generally accompanied by a parallel ability to depress quartz.

PEARL starch, a corn starch prepared with a minimum of alteration, is a good selective depressant for iron oxide minerals.[1] From what is known of the complex chemistry of corn starch, there is evidence that it consists of a mixture of saturated giant polymeric molecules, both straight and branch-chained, the constituents consisting of glucose units with hydroxyl radicals as active or polar groups. To add to the existing knowledge of the chemistry of the flotation process, it is desirable to ascertain the effects on the corn starch as an iron oxide depres-

C. S. CHANG, Junior Member AIME, formerly with the Mines Experiment Station and School of Mines and Metallurgy, University of Minnesota, is now with Chemical Construction Corp., Linden, N. J.

Discussion on this paper, TP 3870B, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript, March 30, 1954.

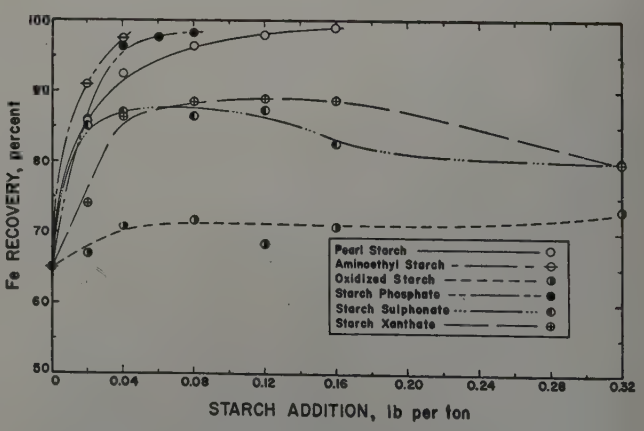


Fig. 1—Effect of starch addition on iron recovery. Laurylamine acetate addition at 0.26 lb per ton.

sant when some of the hydroxyl radicals are replaced by other active groups that are common to flotation reagents.

The ideal way to carry out this investigation would be to test reagents containing different active groups derived from corn starch. The preparation should be such that the reagents possess the same degree and location of substitution and are accompanied by little or no degradation of the starch molecules. This, of course, is almost impossible to realize, as it is difficult to control the degree and location of substitution as well as to prevent degradation of the starch molecule during preparation.

There are two general methods of changing the active group in starch. One is by direct conversion of the active group. The other consists of adding ether or ester groups, containing the desired radicals, to the starch molecule.

Several such substituted starches, derived from a whole corn starch, were prepared in experimental quantities by the Chemical Div., Corn Products Refining Co. Their effects as iron oxide depressants were tested on a wash-ore tailing, laurylamine acetate being used as a collector.

Ore Sample: The iron ore, consisting mainly of hematite, goethite, and quartz, was a wash-ore tailing obtained from the western Mesabi Range. It was

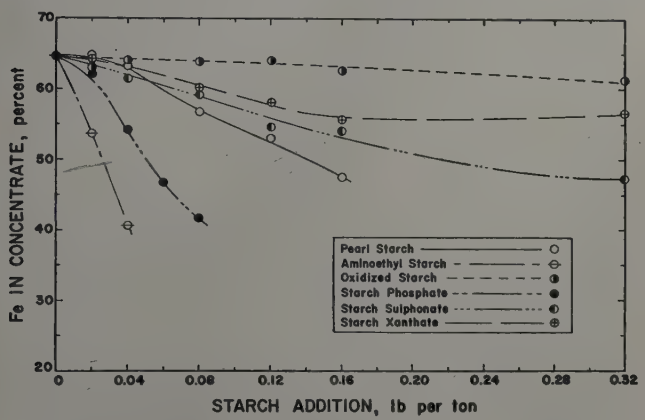


Fig. 2—Effect of starch addition on percent iron in concentrate. Laurylamine acetate addition at 0.26 lb per ton.

ground through 100 mesh, scrubbed with sulphuric acid, and thoroughly deslimed at approximately 20 microns. Flotation feed assayed 33.15 pct Fe.

Flotation: Flotation tests were made in a 50-g pneumatic type cell.[1] The feed was first conditioned for 2 min in 200 ml of solution containing the starch reagents. The collector, laurylamine acetate (99.9 pct pure), was then added and the pulp reconditioned for another 30 sec. The froth was removed until barren of minerals. Demineralized water with impurity less than 0.1 ppm of salts expressed as NaCl was used in all solution preparations. For details pertaining to the ore sample, the method of preparation, and the description of the flotation apparatus, readers are referred to a previous paper.[1]

Starch Reagents: Reactions involved in the preparations of the reagents are illustrated by equations given at the end of the description of each starch.

1—Pearl starch, a whole corn starch prepared with minimum alteration, is essentially insoluble in water at room temperature.

2—Aminoethyl starch, see below, is an ether derivative of corn starch containing, in the sample used, approximately one aminoethyl group ($—O—CH_2CH_2NH_2$) for each 10 glucose units. This material is almost insoluble in cold water.

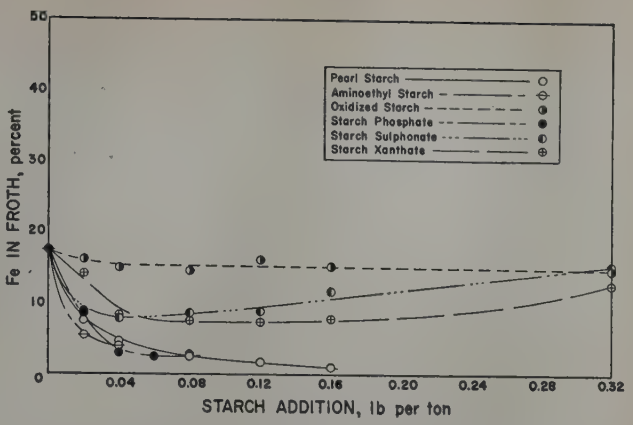
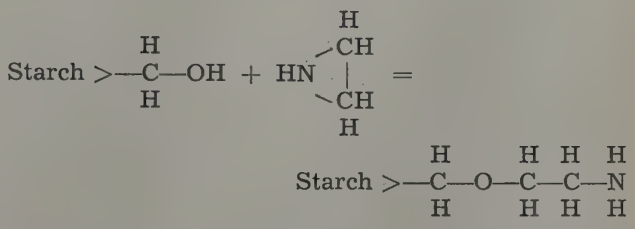
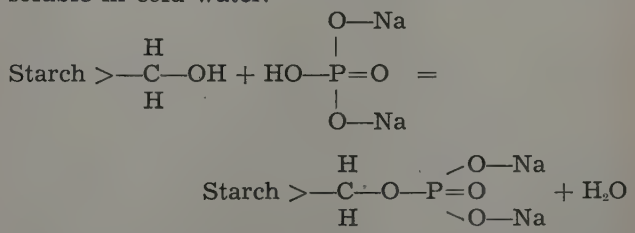


Fig. 3—Effect of starch addition on percent iron in froth. Laurylamine acetate addition at 0.26 lb per ton.



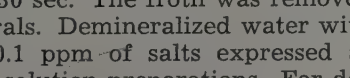
3—Starch phosphate, see below, is an ester derivative of corn starch with the phosphate in the

ortho form. The sample tested was made from a somewhat degraded starch and contained one ester group for approximately each 30 glucose units in the starch molecule. It is insoluble in cold water.

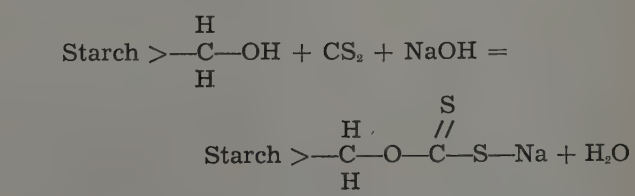


4—Starch sulphonate, a starch derivative prepared by an undisclosed process, contains, in this sample, approximately one sulphonic acid group on a side chain in each 25 glucose units. It also is insoluble in cold water.

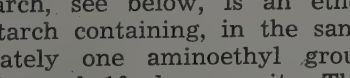
5—Starch xanthate, derived from corn starch,



contains a xanthate group ($—O—C(=S)—S—Na$). The sample used was partially degraded and had one xanthate group in each four glucose units. It is soluble in hot water.



6—Oxidized starch is prepared by treating corn starch with nitrogen dioxide. In the present sample, 35 pct of its terminal hydroxyls are converted to



carboxyl groups ($—C(=O)—OH$). The treatment causes

considerable degradation of the starch molecule and the product is soluble in hot water.

To varying extents these starch reagents may have been degraded during preparation. Also the degree and location of substitution of their active groups is not necessarily the same.

Solutions containing 0.1 pct of these reagents were prepared for flotation addition. The solutions of water-insoluble reagents were prepared by digestion in an autoclave at 120°C for 1 hr. With the exception of pearl starch, which is a common unmodified commercial starch, the reagents used in this study are experimental laboratory products.

Experimental Results

In the presence of a starch depressant, the optimum results for the particular ore sample used were obtained with 0.26 lb laurylamine acetate per short ton of ore.[1] An additional frother is unnecessary, as at this concentration the collector possessed sufficient frothing power. Therefore all flotation tests were made at this same collector addition and the starch addition was varied from 0 to 0.32 lb per ton. The ore was floated at the natural pH of the pulp, 6.5±0.4, since regulating to higher or lower than the natural value in general produced less satisfactory results.

Flotation results are presented in Figs. 1, 2, and 3, which show respectively the iron recovery, the percent iron in the concentrate, and the percent iron in the froth plotted against the starch addition. From these graphs it can be seen that replacement of the active groups in corn starch causes variation in its effectiveness as an iron oxide depressant.

It should be pointed out that the mere addition of 0.26 lb of collector per ton can produce a concentrate assaying about 65 pct Fe with a recovery of about 65 pct, i.e., 35 pct of the iron floats in the silica froth. An ideal selective iron oxide depressant should increase the recovery to almost 100 pct without lowering the iron content of the concentrate.

Oxidized starch has no value as an iron oxide depressant, since it has little or no effect on the separation. Aminoethyl starch and starch phosphate satisfy the recovery requirement, Fig. 1, but the iron content of the concentrate, Fig. 2, declines rapidly with increased addition. This indicates that these two starches, particularly the aminoethyl starch, have a strong tendency to depress not only the iron oxide minerals but also the quartz. This is in agreement with the finding of Cooke and coworkers.[2] Further, the addition of 0.06, 0.10, or 0.20 lb per ton respectively of aminoethyl starch, starch phosphate, and pearl starch depresses all minerals. Although addition of starch sulphonate or starch xanthate does not seriously lower the grade of the iron concentrates neither does it raise the iron recovery to even 90 pct. With increased pearl starch addition the iron recovery rises to almost 100 pct. The increase in recovery is not as rapid as in the case of aminoethyl starch or starch phosphate, but it is not accompanied by the rapid decline of the grade of the iron concentrate. For these reasons a product assaying 60 pct Fe can be obtained with the use of pearl starch at a recovery as high as approximately 95 pct.

Discussion

To become effective as well as selective a depressant must be adsorbed liberally on the mineral to be affected and sparsely, if at all, on the mineral to be floated. This is confirmed by results of an adsorption study made by Schulz and Cooke.[3] Pearl starch coats Brazilian hematite better than it coats quartz. On the other hand, the non-selective aminoethyl starch is adsorbed extensively on both Brazilian hematite and quartz. Mesabi hematite, however, was reported to adsorb less starch per unit of surface than quartz. It must be remembered that the Mesabi hematite used possesses a large internal surface. This is exhibited by the great difference between the air-permeability surface (2060 cm^2 per g) and the krypton-adsorption surface (128,600 cm^2 per g). Since the size of the liquid krypton molecule is much smaller than that of the giant starch molecule, it is possible that the surface accessible to krypton adsorption may be inaccessible to starch adsorption. For this reason the starch adsorption data for Mesabi hematite, which is based on the krypton-adsorption surface, could be several orders of magnitude lower than the true values.

It was also reported by Schulz and Cooke[3] that the addition of Gum 3502 did not affect the adsorption of laurylamine acetate on hematite. This contradicts the general belief that the function of a depressant is to prevent or minimize the adsorption of collector on the mineral surface. Nevertheless it is not difficult to conceive that starch, being of negative charge, is adsorbed simultaneously with the positively charged amine collector by the hematite surface. The fact that the starch molecules are much larger and longer than the amine ion or molecule may very well obscure the hydrophobic characteristics of the collector.

Conclusions

Replacement of active groups in corn starch causes varying effectiveness as an iron oxide depressant in amine flotation of iron ore.

The replacement as shown by the results of the starch derivatives tested impairs rather than improves the value of corn starch as a selective iron oxide depressant.

Increased ability to depress iron oxides is generally accompanied by a parallel ability to depress quartz.

The following starches are listed in the order of decreasing ability to depress iron oxides as well as quartz:

1—aminoethyl starch, 2—starch phosphate, 3—pearl starch, 4—starch sulphonate, 5—starch xanthate, 6—oxidized starch.

Acknowledgments

The author is indebted to Harry Gehman, Assistant Director of Research, Corn Products Refining Co., for his generous contribution, particularly in making available the various starch reagents tested, to V. E. Bye and his associates of the University of Minnesota for conducting the analytical work; and to Strathmore R. B. Cooke of the same institution for his invaluable assistance in preparing this paper.

References

[1] C. S. Chang, S. R. B. Cooke, and R. O. Huch: Starches and Starch Products as Depressants in Amine Flotation of Iron Ore. *Trans.* AIME (1953) **196**, p. 1282.

[2] S. R. B. Cooke, N. F. Schulz, and E. W. Lindroos: The Effect of Certain Starches on Quartz and Hematite Suspensions. *Trans.* AIME (1952) **193**, p. 697.

[3] N. F. Schulz and S. R. B. Cooke: Froth Flotation of Iron Ores; Adsorption of Starch Products and Laurylamine Acetate. *Industrial and Engineering Chemistry* (1953) **45**, p. 2767.

Adsorption of a Mercaptan on Zinc Minerals

by A. M. Gaudin and D. L. Harris

Observations were made of the distribution of mercaptan containing S_{35} between aqueous solution and mineral and between aqueous solution and the gaseous phase. Although equilibrium may not have been attained, adsorption of the reagent was shown to occur readily from air or aqueous solution on sphalerite, zincite, and willemite and to correspond to flotation. Adsorption on quartz did not similarly occur.

THE following results, presented here in condensed form,[1] were obtained in a preliminary study of the adsorption of n-hexane thiol, hexyl mercaptan, on sphalerite, zincite, willemite, and quartz, from aqueous solution and from a gas. Interest in this subject was aroused by a Belgian report[2] of effective use of hexyl mercaptan for flotation collection of oxidized zinc minerals. The relatively low boiling point, 149°C, of the mercaptan[3] suggested the desirability of extending the usual measurements of partition of collector between aqueous solution and gas and between gas and mineral. It is believed that this paper presents the first measurements of this type on a flotation system. Attempts were made to carry out the measurements at equilibrium, but as the work progressed it became increasingly doubtful that this desirable condition had been achieved.

To control composition and extent of the gas phase, the apparatus was a wholly-enclosed thermally-controlled glass system. Because of these constraints and the desirability of dealing with pure minerals, a scale of operations was chosen in which a few grams of deslimed mineral were used in each test. It was also necessary to choose a particularly sensitive method for mercaptan analysis, and in fact a method that would permit the experimenters to follow the approach to equilibrium. For these reasons mercaptan marked by radiosulphur 35 was used. An analysis was made for the radiosulphur by a modification of the method of Gaudin and Carr.[4]

Coarsely-crystallized sphalerite was handpicked, stage-crushed in the dry state, wet-screened on a 200-mesh sieve, and deslimed in water at about 5 microns. Further treatment consisted of a wash in dilute aqueous hydrogen peroxide, drying, removal of the dark-colored fraction in a Frantz magnetic separator, washing in very dilute hydrochloric acid, repeated washing in distilled and conductivity water, and drying. The last washings showed a conductivity equivalent to a few ppm NaCl, that is, much more than would be provided, theoretically, by a saturated ZnS solution. The material was stored dry in sealed bottles. Analyses were as follows: Zn, 62.3 pct; Fe, 0.43 pct; Cd, 0.44 pct; S, 31.2 pct; Mn, 0.001 pct. The specific surface (BET method) was 2000 cm^2/g.

Zincite from Franklin furnace of the New Jersey Zinc Co. was hand-picked, dry-crushed, wet-screened at 100 mesh, and deslimed at about 10 microns. After drying, the associated zinc, manganese, calcium, and silicate minerals were removed in a Frantz magnetic separator. The purified zincite was washed in distilled water and conductivity water to a conductance of less than 2 ppm equivalent NaCl, dried, and stored. Analyses were as follows: Zn, 75.1 pct; Fe, 0.9 pct; Mn, 2.78 pct. The specific surface (BET method) was 1740 cm^2/g.

Willemite, also from Franklin furnace, was purified similarly. Analyses were as follows: Zn, 52.5 pct; Fe, 0.12 pct; SiO_2, 27.3 pct; loss on ignition, 0.13 pct. The specific surface was 1760 cm^2/g.

Conductivity water (double-distilled) and demineralized-distilled water were used in most of the tests. The specific resistance was not less than 600,000 ohms, and usually above 1,000,000.

Radiosulphur-marked hexyl mercaptan (1-hexane thiol) was synthesized by Tracerlab, Inc., Boston. Two lots were secured several months apart. The last lot, consisting of about 0.5 g of the mercaptan, had a total activity of about 10 millicuries. Tracerlab Co. guaranteed only the activity; hence a quasi-vapor pressure determination (based upon an S_{35} analysis) of the mercaptan was made. The calculated value, 4.2 mm of mercury at 25.5° C, has been compared with that of a sample of *Highest Purity* 1-hexane thiol from Fisher Scientific Co. The latter had a vapor pressure of 4.5 mm of mercury at 2.5°C.

Analytical Procedures

The sample containing radiosulphur-marked mercaptan was oxidized to convert the mercaptan sulphur to sulphate, carrier barium sulphate being added to provide a suitable quantity of total barium sulphate in a filter cake. The precipitate was filtered and dried, and counting was carried out either in a streaming-gas (Q-gas) counter for high sensitivity or with an end-window G-M counter for convenience. The oxidized and precipitated mercaptan gave a radioactive count of 65 counts per minute per microgram in the end-window Geiger-Mueller counter and 1100 counts per minute per microgram in a Q-gas counter.

For standardization of the mercaptan solution, 15 replicate analyses were made. The average deviation per measurement was about 1600 cpm in 65,000 cpm, the probable error in the mean being 275 cpm. It

A. M. GAUDIN, Member AIME, is Richards Professor of Mineral Engineering, Massachusetts Institute of Technology, Cambridge, Mass. D. L. HARRIS, Member AIME, is Associate Professor of Mineral Dressing, University of Wisconsin, Madison, Wis.

Discussion on this paper, TP 3871B, may be sent (2 copies) to AIME before Nov. 30, 1954. Manuscript, Jan. 4, 1954.

might therefore be anticipated that a single measurement might be as much as 2 to 3 pct in error. The radiochemical technique is described in detail in the thesis by Dwight L. Harris.[1]

Proportionality of activity measured in a Q-gas flow counter to the amount of mercaptan taken was satisfactory.

Surface area measurements were made in a BET apparatus with Krypton at the temperature of boiling nitrogen.[5]

Experiments in which the partition of mercaptan between aqueous solution and mineral was sought were carried out in a glass vessel of about 200-ml capacity similar to a separatory funnel but with a side arm provided with a fritted glass filter and a stopcock. About 5 g of mineral were placed in this cell, washed with very dilute hydrochloric acid, and washed repeatedly with double-distilled water. Then the vessel was nearly filled with conductivity water and the desired agents were introduced through the stopcock by means of a needle-stem microburette; the mercaptan was added in solution in isopropyl alcohol, as a single addition from a 0.10-ml pipette. The cell was mounted on a rack in a constant-temperature bath and rotated for the selected duration of the experiment at 20 rpm. At the end of the experiment the liquor was drawn through the fritted glass filter and the solution and solid were analyzed for radiosulphur.

This description of the principal test procedure makes it clear that equilibrium was approached from below, that is, that the solid at no time had adsorbed an excess of agent over the equilibrium amount. To approach equilibrium *from above* a few desorption experiments were conducted by exposing agent-covered mineral to the leaching action of a more dilute solution of the agent, or by plain water.

Experiments for the purpose of evaluating the adsorption of mercaptan vapor on dry minerals were semi-quantitative only. Weighed samples of mineral in fritted glass containers and a small amount of active mercaptan in an open cup were placed in a covered jar. The quantity measured was the activity acquired by the mineral (determined by leaching the agent) after the passing of time. Some of the activated mineral was subjected to a moderate vacuum before analysis.

Distribution of mercaptan between aqueous solution and gas phase was obtained for a number of alkaline solutions by two methods. One method was by calculation on the basis of the ionization constant of hexyl mercaptan as measured by Yabroff[3] (1.9×10^{-11}); the measured distribution in neutral solution; the pH of the liquor as measured by the present

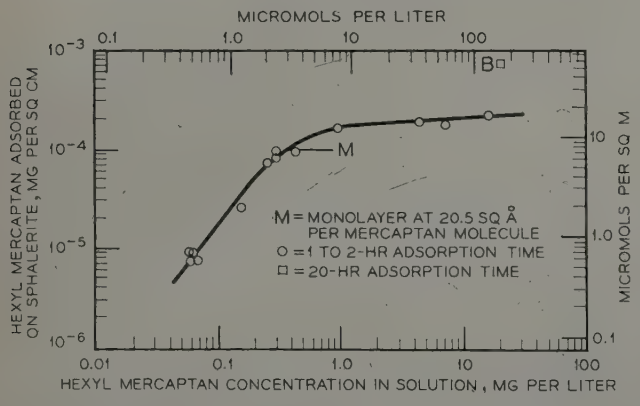


Fig. 1—Mercaptan concentration in solution vs the amount on sphalerite.

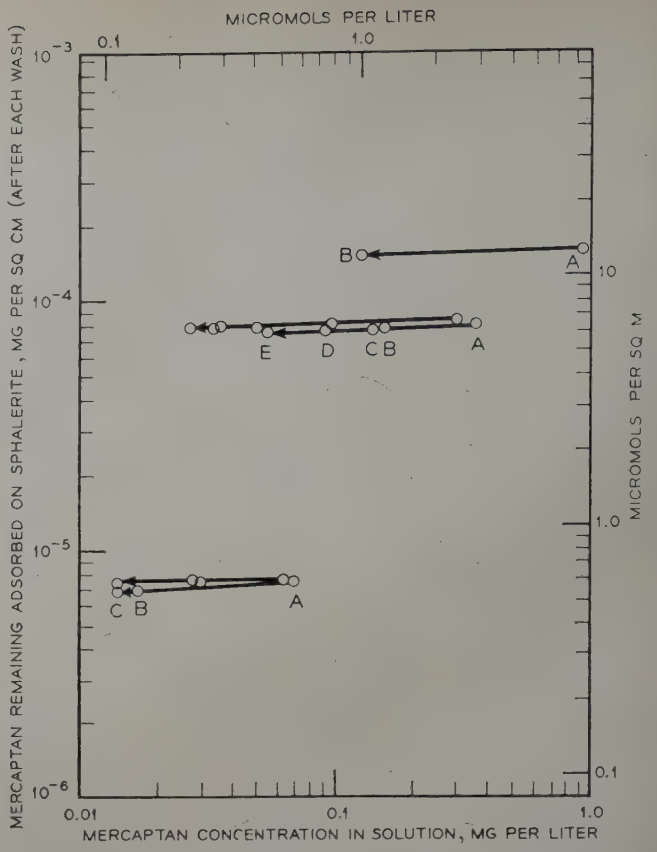


Fig. 2—Desorption. Mercaptan concentration in solution vs amount on mineral. Desorption time equals 1 hr for each succeeding leach.

experiments in a glass-electrode pH meter; the volumes of gas and aqueous solution; and Henry's law. The second method was directly experimental by radiosulphur assay for both gas and liquid phase.

Results from Adsorption Experiments

Sphalerite: Fig. 1 shows the distribution of hexyl mercaptan between sphalerite and aqueous solution (substantially neutral), all measurements representing attempted approaches to equilibrium from below. The hexyl mercaptan analyses were all radiometric, that is, it was implicitly assumed that no radioactive sulphur compounds other than mercaptan occurred in the reagent introduced in the cell or were produced by chemical reaction.

The change of slope at *M* in the region corresponding to monolayer coverage is noteworthy. It is also interesting to note the large increase in activity of the coated mineral on prolonged contact in concentrated solution (point *B*). This suggests that equilibrium was not attained or that side reactions occurred.

Two additional series of experiments were also conducted in the effort to approach equilibrium from above. In one of these series certain of the coated minerals from the experiments reported in Fig. 1 were leached repeatedly with distilled or conductivity water for 1-hr periods and the leaches analyzed. Data for the 5 experiments made are presented in Fig. 2. Successive washes produced increasingly dilute filtrates, as may be seen from the succession of points bearing consecutive letters of the alphabet, but these leachings removed so little S_{35} from the coated mineral as to affect its retention of S_{35} hardly at all. If the data of Fig. 1 had represented equilibrium conditions, desorption data (such as those of

Fig. 2) should have formed a curve identical to Fig. 1 instead of being widely different.

In the second series of desorption experiments much longer periods of equilibration were applied (18 to 50 hr instead of ½ to 2 hr). The data, shown in Fig. 3, differ from those of Fig. 2 in that the assay of the terminal wash water, in at least two cases, was higher than that of the adsorption solution. When the data of Figs. 1, 2, and 3 are taken together, the conclusion must be reached that Fig. 1 does not represent equilibrium conditions or that side reactions occur.

Table I. Adsorption and Desorption of Hexyl Mercaptan on Zincite from Aqueous Solution*

Operation	Time, Hr	Mercaptan Distribution		
		Solution Mg Per Liter	Adsorbed Mg Per Sq Cm	Percentage on Mineral
Adsorption	1.0	0.193	9.0×10^{-5}	96.8
Desorption	1.0	0.155	8.73×10^{-5}	94.3
Desorption	0.5	0.149	8.5×10^{-5}	91.8
Desorption	0.5	0.142	8.3×10^{-5}	89.5
Desorption	0.5	0.123	8.1×10^{-5}	87.5
Desorption	0.5	0.105	7.95×10^{-5}	85.8

* The mercaptan added was 1.0 mg for the test. The pH of the tests was 7.5 to 8.0; the temperature was 26°C; the amount of zincite surface area was 10,800 sq cm; the volume of water was 164 ml for each sorption and desorption test.

One test was conducted to ascertain whether adsorbed radioactive mercaptan, about one monolayer thick, would exchange with inactive mercaptan in solution. This test showed that about 0.8 pct of the radiosulphur was emitted in 1½ hr. It may be concluded that under the experimental conditions used the rate of exchange is extremely slow, that the equilibrium concentration of true mercaptan in solution is extremely minute, and/or that side reactions interfere with exchange.

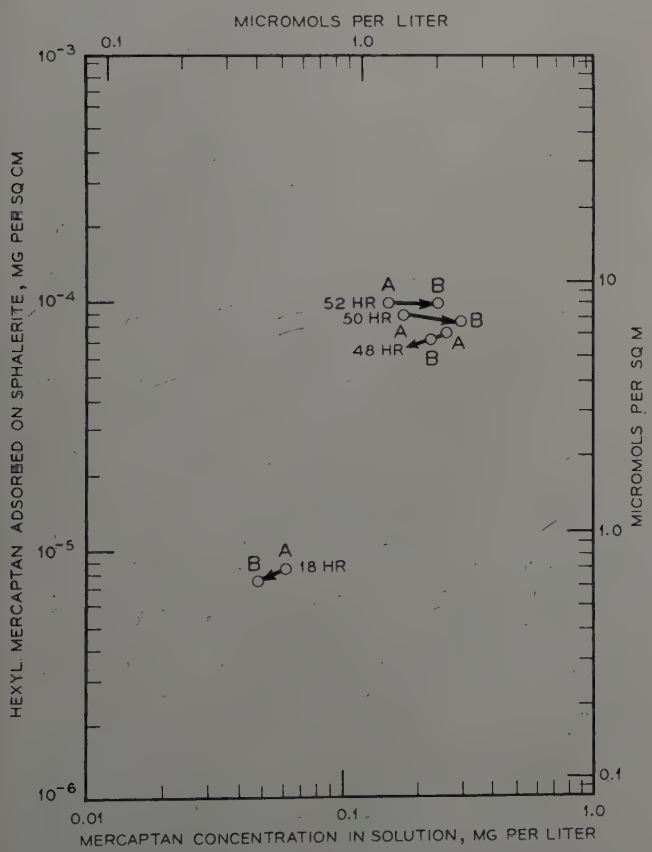


Fig. 3—Desorption. Mercaptan concentration in solution vs amount on sphalerite. Long desorption periods.

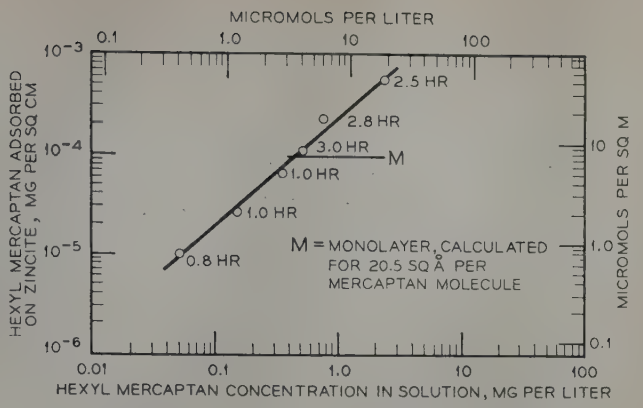


Fig. 4—Mercaptan concentration in solution vs the amount on zincite.

Several tests were made in alkaline circuits ranging from pH 7.5 to pH 12. The data suggest that pH within this range is not an important variable. An increase in temperature up to 40°C also had no significant effect.

Zincite and Willemite: Fig. 4 shows the data obtained on the system zincite-water for mercaptan distribution (equilibrium approached from below). The data show almost linear dependence of the amount adsorbed on the concentration of the mercaptan, with no saturation in sight when a monolayer is reached.

A single adsorption-desorption test was made to determine whether equilibrium was attained. The results indicate, see Table I, that adsorbed mercaptan is removed with difficulty and that equilibrium probably was not attained.

Data reporting the mercaptan distribution in the water-willemite system are presented in Table II.

Table II. Adsorption of Hexyl Mercaptan from Aqueous Solution Onto Willemite*

Test No.	Mercaptan Added in Cell	Agitation Time, Hr	Mercaptan Analyses		Mercaptan Abstracted by Solid Pct of Total
			Solution Concentration, Mg Per Liter	Abstracted by Mineral Mg Per Sq Cm	
1	0.1	2	0.133	0.82×10^{-5}	78.2
2	0.1	1.5	0.156	0.78×10^{-5}	74.4
3	1.0	2	1.66	7.66×10^{-5}	72.7
4**	1.0	2.1	2.64	8.17×10^{-5}	77.8
5	2.0	2	2.58	16.5×10^{-5}	78.2
6	10.0	3.7	21.8	67.5×10^{-5}	64.3

* Temperature = 26°C; pH = 7.5 approximate; solution volume = 164 ml; mineral sample = 5.4 g; total mineral surface area 9500 sq cm.

** Test 4 was made with a gas phase of 80 ml and a liquid phase of 84 ml. The mercaptan in the gas phase represented only 0.88 pct of the total added to the cell.

This table shows that mercaptan is readily adsorbed and also that the solution concentration represents a larger proportion of the agent added to the cell than in the cases of zincite or sphalerite.

Indirect measurements (the analyses of a gas phase in equilibrium with the solution and mineral phase, and a comparison with distributions measured for hexyl mercaptan in solution and gas phases) suggested that most of the sulphur (the basis of the mercaptan analysis) in the filtrate was not present as hexyl mercaptan.

Quartz and Pyrex Glass: Tests were made to ascertain whether quartz and pyrex powders adsorb mercaptan from neutral aqueous solution. They showed no measurable adsorption.

Adsorption From the Gas Phase: Semi-quantitative tests are reported in Table III. Mercaptan

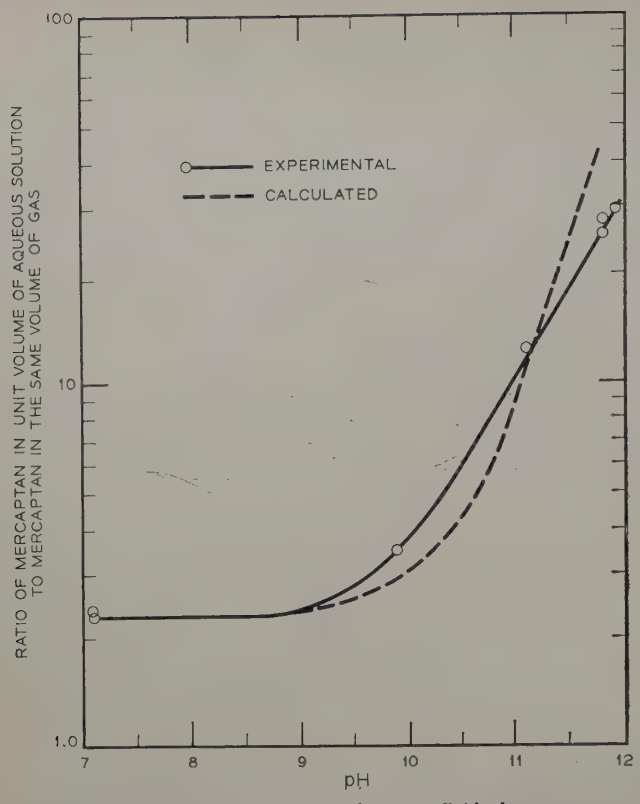


Fig. 5—Distribution of mercaptan between fluid phases as a function of alkalinity.

vapor at room temperature, 28° to 34°C, in an air atmosphere was adsorbed on dry samples of mineral and the solids were analyzed. The results, tests 1 to 5, showed that mercaptan vapor is readily taken up by sphalerite. Test 5 also indicates that this amount (equivalent to several monolayers) is strongly adherent; after evacuation for 7.5 hr the amount of mercaptan retained by the mineral is approximately the same.

Table III. Adsorption of Hexyl Mercaptan Vapor on Dry Mineral*

Mineral	Test No.	Time, Days	Mercaptan Adsorbed, Mg Per Sq Cm
Sphalerite	1	4	2.54 X10⁻⁴
	2**	4	2.49 X10⁻⁴
	3	4	4.5 X10⁻⁴
	4	7	3.2 X10⁻⁴
	5†	7	2.8 X10⁻⁴
Monolayer at 22.5 A²/RSH molecule =			0.87 X10⁻⁴
Zincite	6	4	6.5 X10⁻⁴
	7	7	14.5 X10⁻⁴
	8†	7	2.8 X10⁻⁴
Willemite	9	4	4.7 X10⁻⁴
	10	7	3.1 X10⁻⁴
Quartz	11	7	0.19 X10⁻⁴
	12†	7	0.07 X10⁻⁴

* Conditions were identical for Tests 1 and 2, identical for Tests 4, 5, 7, 8, 10, 11, and 12 and identical for Tests 3, 6, and 9.
** Pulped in water and 3.28 pct of total mercaptan removed in the water, and 0.7 pct of total mercaptan removed on a fine fritted glass filter.
† Evacuated 7.5 hr before leaching.

Mercaptan is readily adsorbed also on dry zincite. The amount taken up in 7 days reaches the equivalent of 16 monolayers (test 7). Evacuation of a coated sample reduces the adsorption to about three monolayers, an amount equivalent to that retained by sphalerite after evacuation (test 8).

Two tests (9 and 10) are reported for the adsorption of mercaptan vapor on willemite. The amount of mercaptan taken up is comparable to that on sphalerite, about three monolayers in 4 to 7 days. For comparison with a nonreactive mineral, results are presented for adsorption of mercaptan vapor on quartz. Tests 11 and 12 show that the quartz does not effectively take up mercaptan.

Experiments of the same general type, refined to become truly quantitative, should open a large new field for understanding of flotation mechanism.

The theoretical method of evaluating the quantity of mercaptan in the gas phase in equilibrium with aqueous solutions of various basic pH values was based on several quantities, i.e., the amount of mercaptan added to the closed system, the concentration at the respective pH values, the distribution or mercaptan between gas and aqueous solution at a neutral pH, the ionization constant, and the application of Henry's law. Qualitative accord is noted between calculated values and measured values in Fig. 5.

Qualitative flotation and agglomeration tests were made for the zinc minerals, sphalerite, zincite, and willemite and for quartz powder as a comparison. In those partition tests in which a gas phase occurred together with the solution and mineral phases, agglomeration was readily noted for the zinc minerals after adsorption of mercaptan corresponding to less than monolayer coverage. Qualitative flotation tests in a test tube were also made with the application of vacuum to indicate flotation of the zinc minerals. These tests showed that adsorption of the mercaptan from the vapor phase prior to flotation testing was effective in causing flotation. On the other hand, quartz powder was not induced to float, or agglomerate, under similar conditions after having been exposed to mercaptan in the vapor form and/or in aqueous solution.

Summary

Adsorption of hexyl mercaptan on sphalerite, zincite, and willemite was shown to occur readily from water or from air but was shown to be absent for quartz. Qualitative flotation tests proved that flotation of the minerals occurred even when the amount taken up was equivalent to less than a monolayer. However, several effects are noted which suggest that equilibrium conditions have not been obtained in the adsorption and desorption tests in aqueous media, or that the mercaptan was impure.

Acknowledgements

The authors wish to thank Professors J. Th. G. Overbeek, P. L. de Bruyn, and D. W. Fuerstenau for their unstinting help. They also express appreciation to the U. S. Atomic Energy Commission for making this research possible and to the New Jersey Zinc Co. for supplying mineral products used in the research.

References

[1] Dwight L. Harris: Hexyl Mercaptan and Zinc Minerals. ScD. Thesis, Department of Metallurgy, Massachusetts Institute of Technology, 1953.

[2] F. Gaty, H. de Rycker, and H. Thyssen: Étude de la Flottation des Minerals Calaminaires aux Laboratories de l'Institut de Metallurgie. *Révue Universelle des Mines de la Metallurgie* (Belgium, 1946) **89**, pp. 153-166.

[3] D. L. Yabroff: Extraction of Mercaptans with Alkaline Solutions. *Industrial and Engineering Chemistry* (1940) **32**, p. 257.

[4] A. M. Gaudin and John S. Carr: Analytical Procedures for Ethyl Xanthate Labeled with Radiosulphur. *Analytical Chemistry* (1952) **24**, pp. 887-889.

[5] F. W. Bloecher, Jr.: A New Surface Measuring Tool for Mineral Engineers. *Mining Engineering* (1951) **3**, p. 255.

Safety Factor Characteristic Curves

by W. A. Boyer

Their Application to Mine Hoisting Ropes

If the safety factor of a mine hoisting rope is checked for the lowest depth, is the rope then safe for all levels? The answer here is no. A new set of values is proposed.

HOISTS for metal mines are seldom designed for one particular depth. They are intended for an ultimate load and depth with a given speed but are first used to hoist from a shallower depth, gradually working to the ultimate as development progresses. Sometimes the mine is not worked on the upper levels because of the kind and grade of ore, the mining of which depends on market conditions. In this case the hoist may be used in the same working shift to lift ore, for example, from the 1200 and the 4400-ft level.

This means that the safety factor of the rope should be considered not only when it hoists from the greatest depth, but also when it hoists from the upper levels. According to present safety factor standards, a hoisting rope can have ample safety margin when hoisting from the lower or lowest level and still be shy of sufficient safety margin when hoisting from the upper levels. This will be shown by curves presented in this paper.

The safety factor of a hoisting rope as defined by the U. S. Bureau of Mines[1] is the ratio of the static load to the tensile strength of the rope. It is calculated by dividing the breaking strength of the rope*

* As stated by the manufacturer or as determined by testing.

by the sum of the maximum weights to be hoisted, including the weight of the skip or car, the weight of the material to be hoisted, and the total weight of the rope when it is extended to the bottom of the shaft. The safety factors recommended by the U. S. Bureau of Mines are presented in Table I.

W. A. BOYER, Member AIME, is Superintendent, Mechanical and Electrical Depts., Northwest Mining Div., American Smelting & Refining Co., Wallace, Idaho.

Discussion on this paper, TP 3875A, may be sent (2 copies) to AIME before Dec. 31, 1954. Manuscript, March 25, 1954. Meeting Northwest Mining Assoc., Spokane, Wash., Dec. 5, 1953.

In Fig. 1 curves *A* and *B* are plotted against the vertical suspended distance, which is the abscissa, and the factor of safety, the ordinates. Curve *C* is a smooth curve drawn through the midpoints of the steps of curve *A* and would more closely represent the minimum factor of safety for new rope for any particular depth. Curve *D* is a smooth curve drawn through the midpoints of the steps of Curve *B* and would more closely represent the minimum factor of safety when ropes should be discarded for any particular depth. If a new rope were applied with a minimum factor of safety, then the vertical distance or ordinate under curve *D* for any particular depth, divided by the vertical distance or ordinate under curve *C*, would represent the minimum percent of remaining area intact that could be allowed before the rope would be discarded.

Table I. Hoisting Rope Safety Factors As Defined by the U. S. Bureau of Mines

Length of Rope, Ft	Minimum Factor of Safety for New Rope	Minimum Factor of Safety When Rope Must Be Discarded
500 ft or less	8	6.4
500 to 1000 ft	7	5.8
1000 to 2000 ft	6 (curve *A*)	5.0 (curve *B*)
2000 to 3000 ft	5	4.3
3000 ft and more	4	3.6

Also, that section of the ordinate between curve *C* and curve *D* is proportional to the section of the area of the new rope that could be allowed for wear and broken wires. Considering that point at 2500 ft on the chart, the ratio *do* to *co* would be the minimum percent of rope area remaining at the point of discard. The ratio of that portion of the ordinate *cd* to *co* would represent maximum percent of area of a new rope available for wear and broken wires.

This page of Mining Transactions AIME follows p. 928. The intervening non-Transactions pages appeared in Mining Engineering.

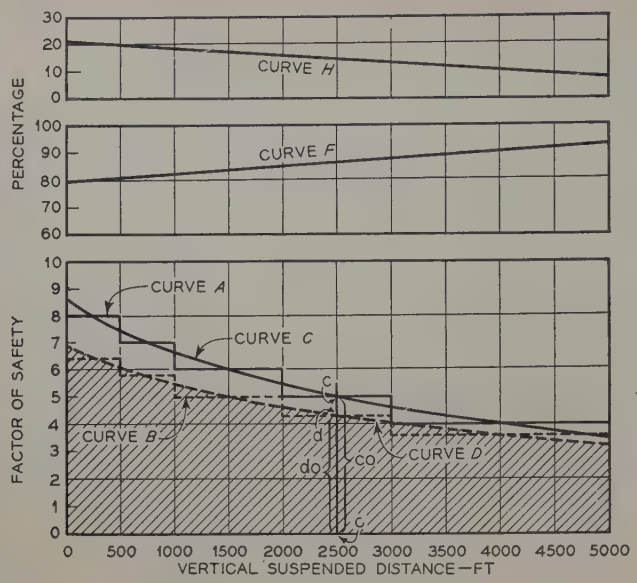


Fig. 1—A study of old factor of safety standards with ropes calculated for the maximum loading or the minimum factor of safety.

Curve *A*—Minimum factor of safety for new rope (U.S. Bur. Mines).
Curve *B*—Minimum factor of safety when rope must be discarded (U.S. Bur. Mines).
Curve *C*—Smooth curve drawn through midpoints of steps of curve *A*.
Curve *D*—Smooth curve drawn through midpoints of steps of curve *B*.
Curve *F*—Minimum percent of remaining area intact at point of discard.
Curve *H*—Maximum percent of area of new rope available for wear and broken wires.

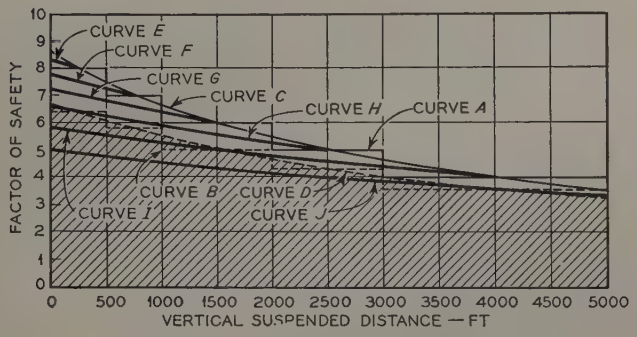


Fig. 3—Comparison of old safety factor curves with characteristic curves of modern rope with various loadings. Shaded area is that portion below curve *D* when rope must be discarded, present standard.

Static loading: 1¼-in. improved plow steel rope. Weight of rope, lb per ft: 2.50. Breaking strength: 64.6 tons or 129,200 lb. Acceleration rate: 0.

Curve *A*—Minimum factor of safety for new rope, old standard.
Curve *B*—Minimum factor of safety when rope must be discarded, old standard.
Curve *C*—Curve drawn through midpoints of steps of curve *A*.
Curve *D*—Curve drawn through midpoints of steps of curve *B*.
Curve *E*—Characteristic curve for 1¼-in. improved plow steel rope at maximum loading, 250 ft.
Curve *F*—Characteristic curve for 1¼-in. improved plow steel rope at maximum loading, 750 ft.
Curve *G*—Characteristic curve for 1¼-in. improved plow steel rope at maximum loading, 1500 ft.
Curve *H*—Characteristic curve for 1¼-in. improved plow steel rope at maximum loading, 2500 ft.
Curve *I*—Characteristic curve for 1¼-in. improved plow steel rope at maximum loading, 4000 ft.
Curve *J*—Characteristic curve for rope of curve *I* when ready for discard under requirements of old standard.

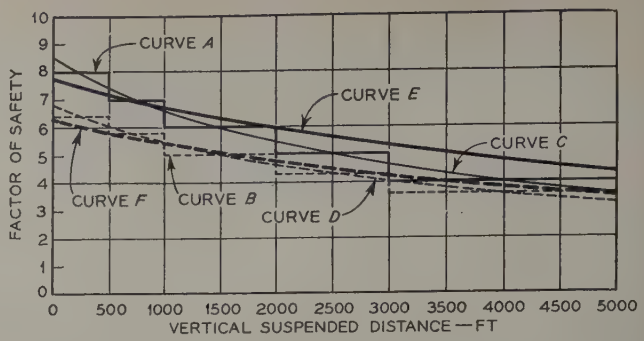


Fig. 2—Comparison of old factor of safety values and new proposed factor of safety values.

Curve *A*—Minimum factor of safety for new rope, present standard.
Curve *B*—Minimum factor of safety when rope must be discarded, present standard.
Curve *C*—Smooth curve drawn through midpoints of steps of curve *A*.
Curve *D*—Smooth curve drawn through midpoints of steps of curve *B*.
Curve *E*—Minimum factor of safety for new rope, new proposed standard.
Curve *F*—Minimum factor of safety when rope must be discarded, new proposed standard.

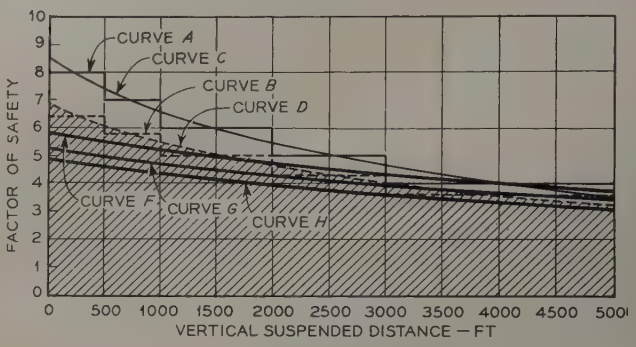


Fig. 4—Comparison of an actual rope safety factor characteristic curve with U. S. Bureau of Mines values. In this instance the rope is loaded under static conditions to the value of 4 at 4000 ft. Shaded area is that portion below curve *D* when rope must be discarded.

Weight of skip, cage, and ore: 22,300 lb. Size of rope: 1¼ in. Grade of steel in rope: improved plow steel, 6x19. Weight of rope, lb per ft: 2.50. Breaking strength of rope: 64.6 tons or 129,200 lb.

Curve *F*—Acceleration: 0, static loading.
Curve *G*—Acceleration: 0.10 g or 3.216 ft per sec per sec.
Curve *H*—Acceleration: 0.20 g or 6.432 ft per sec per sec.

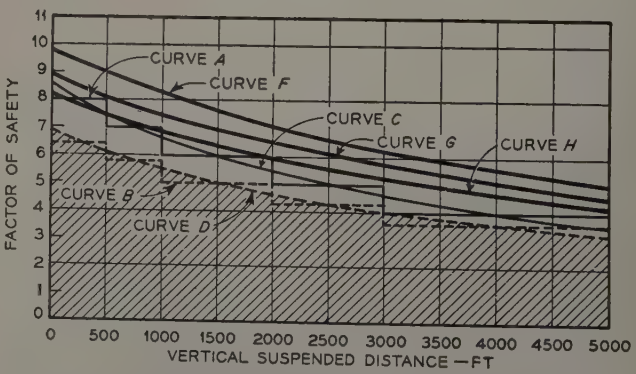


Fig. 5—Compare with Fig. 4. Shaded area is that portion below curve *D* when the rope must be discarded.

Weight of skip, cage, and ore: 13,206 lb. Size of rope: 1¼ in. Grade of steel in rope: improved plow steel, 6x19. Weight of rope, lb per ft: 2.50. Breaking strength of rope: 129,200 lb.

Curve *F*—Acceleration: 0, static loading.
Curve *G*—Acceleration: 0.10 g or 3.216 ft per sec per sec.
Curve *H*—Acceleration: 0.20 g or 6.432 ft per sec per sec.

If these ratios for the different depths are worked out and plotted, curve *F* represents the minimum percent of remaining area intact at point of discard for any particular depth, and curve *H* the maximum percent of area of the new rope available for wear and broken wires. Checking these curves reveals that the factor of safety schedule as set up allows for a 10 pct loss of area of the new rope to cover wear and broken wires at 4500 ft and would allow a 20 pct loss of area of the new rope to cover wear and broken wires at 250 ft. By the above standards the point of discard of a given rope would be determined by the allowable percent for wear and broken wires for the greatest depth from which the rope works.

Curves *C* and *D* of Fig. 1 do not follow along the characteristic curve of any rope of modern construction used for hoisting purposes. They do exactly coincide with the characteristic curve of a 1¼-in. mild steel rope having a breaking strength of 75,000 lb.** The characteristic curve of the present 1¼-in. plow steel rope, which has a breaking strength of 129,200 lb, is much flatter when loaded for the safety factor values used here.

** Such a rope was listed in a manufacturer's catalogue of 1913.

It is here proposed that new safety standards be set up governed by the characteristic curve of a rope of modern construction and material. It is also suggested that a 20 pct reduction of effective area of the rope be allowable at all depths for wear and broken wires. On the basis of these new standards a pound loading can be given for each size of rope which automatically will take care of the safety factor requirements. Table II shows the old and the new proposed values for the factor of safety for various depths. The old values given are taken from curves *C* and *D* as being more comparable.

Old and new values for the factor of safety are plotted in Fig. 2. It will be noted that above 900 ft the old values are higher than the new but that below 900 ft the new values become increasingly higher with increase in depth.

In Fig. 3 curves are plotted for 1¼-in. improved plow steel rope according to the old factor of safety standards and according to the new standards providing an allowable factor of safety for the various maximum depths as indicated. Perhaps no greater proof is needed of the inadequacy of the older method. In all cases the rope characteristic curve is much flatter. Take, for example, the rope calculated for 4000 ft, curve *I*. The new rope would not fulfill the factor of safety requirements for a rope to be discarded above the 1500 ft point. Curve *J* is the characteristic curve for the rope of curve *I* with a reduction of effective area of 10 pct or for the point of discard at 4000 ft. This allows the use of a rope for depth which falls far short of fulfilling the factor of safety requirements for the shallower depths. The author cannot see that the present standards are accomplishing their aim when such conditions are possible. If the engineer checks the factor of safety for a given rope application for the upper levels and lets that govern the size of the rope he is unconsciously following the new method proposed here.

The margin established by the U. S. Bureau of Mines is the static or *dead load* factor of safety. A discussion of the old safety factor standards published in 1923[2] states that the acceleration stresses should be kept out of the factor of safety calculations. On the contrary, acceleration stresses should be included. Figs. 4, 5, 7, and 9 show the effect of acceleration on factor of safety values.

An *operating* factor of safety is recommended which will include the stress added by acceleration forces. The additional stress due to an acceleration rate of 3.216 ft per sec per sec, or 0.10 g, will increase the overall stress on a rope by 10 pct and lower the static factor of safety by nearly 9.1 pct. If the factor of safety for 1¼-in. plow steel rope is 5.55 at 2500 ft, the total connected load at the end of the cable would be reduced by 2113 lb with the inclusion of acceleration stress from an acceleration of 3.216 ft per sec per sec. Some acceleration and deceleration rates are higher than the value mentioned, so that this additional force can make an appreciable difference in the actual factor of safety of the hoisting rope. This is shown in Fig. 4.

The plotting of a safety factor characteristic curve for a given hoisting rope is as follows. The *operating* stress is first calculated for different depths, at points close enough together to give a smooth and consistent curve. These points are then plotted on a graph on which curves *A*, *B*, *C*, and *D* are already plotted and a curve is drawn through the points. It can then be seen at a glance how the particular rope application compares with the standard for all sections of the shaft.

Fig. 4 illustrates what is probably an extreme condition, but one allowed by the present factor of safety requirements. The safety factor characteristic curve for a 1¼-in. improved plow steel rope is plotted as curve *F*. The rope is loaded so that the factor of safety at 4000 ft is 4, the minimum allowed by the table for static loading. A comparison of curve *F* with curve *A* shows that the application could be allowed for any distance between 3000 and 4000 ft; a comparison of curve *F* with curve *C* reveals that it could not be used for anything above 4000 ft; and a comparison of curve *F* with curve *D* indicates that above the 1500 ft point the factor of safety requirements for the new cable would not equal even the point of discard standard. Inclusion of any acceleration stress lowers the factor of safety so that the rope should not be considered in any case.

Table II. Comparison of Old and New Values for the Safety Factor for Various Depths

Length of Rope, Ft	Minimum Factor of Safety for New Rope		Minimum Factor of Safety When Rope Must Be Discarded	
	Old	New	Old	New
0	8.57	7.75	6.92	6.19
500	7.5	7.18	6.21	5.74
1000	6.67	6.69	5.63	5.35
1500	6.0	6.26	5.14	5.01
2000	5.45	5.88	4.74	4.71
2500	5.0	5.55	4.39	4.44
3000	4.62	5.25	4.09	4.21
3500	4.29	4.98	3.83	3.99
4000	4.00	4.74	3.6	3.8
4500	3.75	4.52	3.4	3.63
5000	3.49	4.32	3.22	3.47

It should be noted that with this low factor of safety or high unit stress in the rope the *safety factor characteristic curve* for the rope is much flatter than the minimum required curve, *C*. If the unit stress in the rope is lessened or the factor of safety increased, the characteristic curve becomes steeper. The use of better grades of steel in rope results in flatter safety characteristic curves.

In Fig. 5 the loading for the 1¼-in. improved plow steel rope has been lessened, or the factor of safety increased, so that under static loading and at 4000 ft the factor of safety is 5.6. Curves *G* and *H* are calculated with the same loading but include ac-

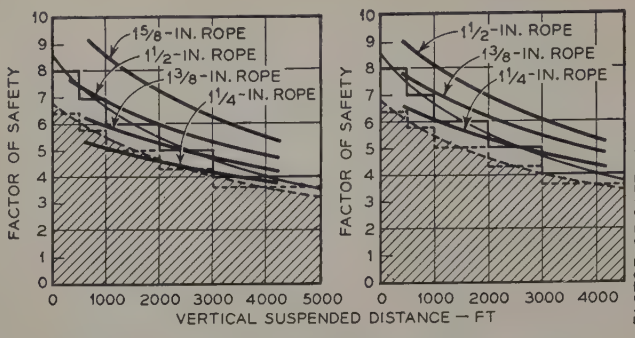


Fig. 6a (left) and Fig. 6b (right)—Studies of rope requirements for a new installation. Note that the calculated factors of safety are operating, not static. All ropes are made of improved plow steel.

At left, capacity: 1000 tons in 12 hr hoisting.

Rope speed: 1200 fpm.
Weight of cage: 3800 lb.
Weight of skip: 6500 lb.
Weight of ore: 10,200 lb.
Maximum depth: 4000 ft.

At right, capacity: 1000 tons in 12 hr hoisting.

Rope speed: 1500 fpm.
Weight of cage: 3600 lb.
Weight of skip: 4800 lb.
Weight of ore: 8350 lb.
Maximum depth: 4000 ft.

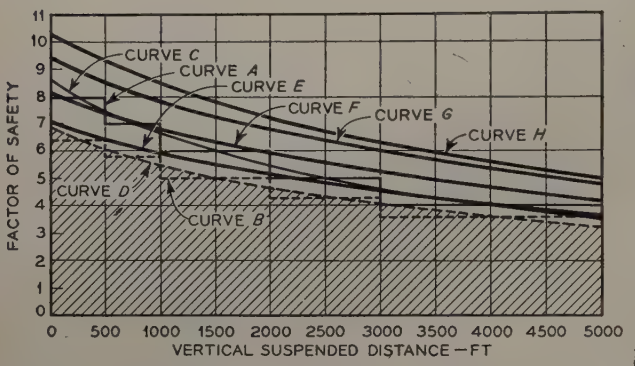


Fig. 7—Comparison of various grades of steel ropes to particular installation. Load is selected to load the 1¼-in. mild plow steel rope to a factor of safety of 4 at 4000 ft, equal to 13,206 lb. The mild steel, plow steel, and the improved plow steel ropes to be made of 6x19 or 6x21 construction and the flattened strand rope to be made of improved plow steel wire.

Weight of cage, skip, and ore: 13,206 lb for all ropes. Acceleration rate to be 1.608 ft per sec per sec.

Curve *E*—Mild plow steel rope, 1¼ in.; weight per ft, 2.50 lb; 97,600 lb breaking strength.
Curve *F*—Plow steel rope, 1¼ in.; weight per ft, 2.50 lb; 112,400 lb breaking strength.
Curve *G*—Improved plow steel rope, 1¼ in.; weight per ft, 2.50 lb; 129,200 lb breaking strength.
Curve *E*—Flattened strand rope, 1¼ in.; weight per ft, 2.81 lb; 142,000 lb breaking strength.

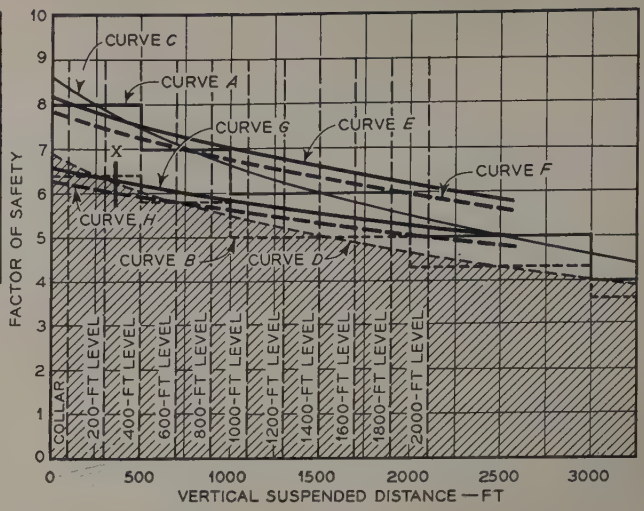


Fig. 8—Characteristic curves of rope used on 400-hp electric hoist. Shaded area is that portion below curve *D* when rope must be discarded.

Conditions under which hoist was designed.

Weight of skip and cage: 8740 lb.
Weight of ore: 7120 lb.
Size of rope: 1⅛ in.
Speed of hoist: 1200 fpm.
Maximum depth: 2000 ft.

Conditions under which hoist was used.

Weight of skip and cage: 8000 lb.
Weight of ore: 5000 lb.
Size of rope: 1⅛ in.
Speed of hoist: 1200 fpm.
Maximum depth: 2000 ft.

Curve *E*—Characteristic curve for rope, static, designed conditions.
Curve *F*—Characteristic curve for rope, operating, designed conditions.
Curve *G*—Characteristic curve for rope, static, actual working conditions.
Curve *H*—Characteristic curve for rope, operating, actual working conditions.

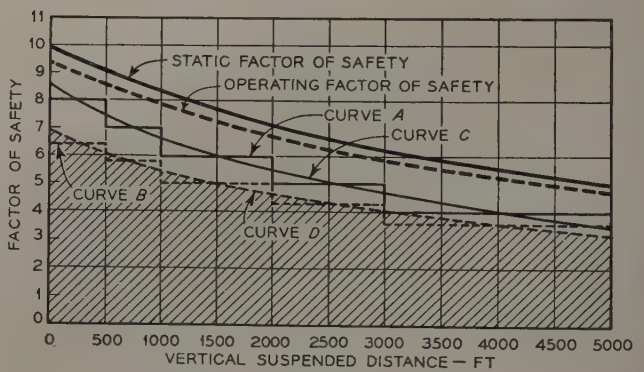


Fig. 9—Characteristic curve of rope used on 2150-hp double-drum electric hoist. Shaded area is that portion below curve *D* when rope must be discarded.

Weight of skip and cage: 14,500 lb.
Weight of ore: 14,000 lb.
Size of rope: 1⅞ in. improved plow steel.
Weight of rope: 5.63 lb per ft.
Breaking strength of rope: 141 tons or 282,000 lb.
Acceleration rate: 1.875 ft per sec per sec.

celeration, as was done in Fig. 4. These curves show, even with the lighter loading, the effect of acceleration stress on the factor of safety of a hoisting rope. With lessening of load on any given cable, safety factor characteristic becomes steeper.

When specifications for a new hoist are being considered, usually the only definite information to work from is the total tonnage desired in so many hours hoisting from a certain maximum depth. Fig. 6a presents two sets of curves worked out for a particular installation, each set indicating how various rope sizes fit into the application with a given rope speed. Fig. 6b presents a set of curves for the same installation but for a different hoist speed.

Even a casual study of these curves leaves no question as to the size of rope that would be necessary for each speed. If the factor of safety for the rope selected is high, the curves will indicate the additional pay load that can be added within the safety limits for the full length of the shaft.

Fig. 7 shows how ropes made with different grades of steel fit into the picture. In this case both the ac-

celeration rate, 1.608 ft per sec per sec, and the combined weight of cage, skip, and ore, 13,206 lb, are the same for each rope. The curves show that ropes of higher steel strength increase the safety factor. The *flattened strand* rope is of the same grade of steel as the improved plow steel rope but of different stranding construction. It is interesting to note that at the 4000 ft point there is a 15 pct increase in safety factor when the plow steel rope is used instead of the mild plow steel and also a 15 pct increase in safety factor when improved plow steel is used instead of plow steel. Although the flattened strand is approximately 10 pct stronger than the improved plow steel, it weighs 12.4 pct more per ft and therefore adds only 4.53 to the safety factor value.

In the example given above the flattened strand rope does not show up advantage as it does if the allowable connected load using a constant safety factor of 5.55 at 2500 ft is compared. When this comparison is made, the allowable connected load of improved plow steel is 22.5 pct over that of plow steel and the allowable connected load for flattened strand rope 9.1 pct over that of improved plow steel.

Setting aside other advantages there might be in using flattened strand rope rather than improved plow steel for a new installation, its superior strength is not sufficient to justify the additional cost. However, when the rope factor of safety is slightly low in an old installation, or when it is desirable to increase the payload of the hoist and still maintain the former margin of safety, flattened strand rope can be used to advantage.

Safety factor characteristic curves for two typical hoisting applications are shown in Figs. 8 and 9. Fig. 8 shows the curve for a 400-hp double-drum electric hoist originally designed to take 1⅛-in. rope while hoisting a total connected load of 13,000 lb from a maximum depth of 2000 ft. At this particular installation there were times when an extremely high-grade galena ore was hoisted, the weight of the ore per cubic foot being considerably higher than the value used in the design of the skip. When this occurred the rope was then operating on the factor of safety curve shown as curve *I*. After about 5 years of operation the hoist was halted by the emergency stop when the upcoming skip was about 400 ft from the sheave, or point x on the curve, and the rope broke. At this point, with the actual loading, a new rope would not fulfill the conditions even at the point of discard. Immediately after the accident, the rope was changed to 1¼-in. improved plow steel and the weight of the skip, cage, and ore reduced, so that the factor of safety at all times was maintained at a higher level.

It is general practice to check the safety factor for maximum depth only, no thought being given to probable increase in the density of ore hoisted. The above illustration, which shows the fallacy of such practice, indicates that plotting the safety factor characteristic curve is needed as a rapid means of determining the fitness of a given rope for a particular application.

Fig. 9 shows the safety factor characteristic curve for rope installed on a large electric hoist for a mining company in the West. This curve was calculated from information published in one of the mining journals. The double-drum electric hoist is driven by a 2150-hp dc motor. The load consists of the weight of skip and cage, 14,500 lb, and the weight of ore, 14,000 lb. The rope used is 1⅞-in. improved plow steel operating at 2300 ft per min from a maximum depth of 5200 ft. According to the characteristic curve for the operating safety factor, which is 5.22 at 4000 ft, the allowable wear area of the rope is near 33 1/3 pct for the full length of the shaft.

Comparison of a number of characteristic curves reveals that the safety factor characteristic curve for a given grade of rope at the same rate of acceleration, starting with the same factor of safety, for example, 5.55 at 2500 ft, will hold very close to the same line for all sizes of rope for that grade. The variation is only 0.22 for zero length of rope and 0.06 for 5000 ft of rope between the sizes of ⅝ in. and 2¼ in. for improved plow steel. It is therefore possible to draw safety factor characteristic curves for any particular grade of rope with different amounts of loading and with various rates of acceleration; the same family of curves would be applicable for all sizes of rope of the same grade, such as improved plow steel.

In conclusion, these points should be considered:

1—In this paper values have not been questioned for the minimum factor of safety for the point of discard. It is suggested, however, that employing a smooth curve instead of the step curve will lessen confusion at the vertical jogs in the line which might raise the question as to what value to use. It is also recommended that values for the various depths be changed slightly so that they fall along the inherent safety factor characteristic curve for an improved plow steel rope with a breaking point of 80 pct of that proposed for the new rope. Although the old values set up some 30 years ago appear to have stood the test of time, to match the progress in metallurgy and manufacture of steel hoisting ropes a re-evaluation should be made by a committee.

2—It is suggested that the minimum factor of safety for new ropes be set up as a smooth curve, the slope of which would closely approximate that of improved plow steel rope. It would then be possible to assign allowable load values to each size of rope and grade and rate of acceleration. If these load values were not exceeded the factor of safety for the rope would automatically take care of itself without further calculation.

3—The factor of safety should include the stress of acceleration, that is, the factor of safety should be an operating value.

4—Consideration should be given the rates of deceleration during emergency stops, which can be made at any portion of the shaft. It is believed that on some ac-powered hoists rate of deceleration may go as high as 1.0 g or more. In such cases, stress on the rope on the down-going skip side, although the skip is empty, would be considerably greater than when a loaded skip is accelerated. A closer definition of maximum stress on a rope is also valuable in determination of maximum stress in members of skips and cages.

5—To prolong the life of a rope, the safety factor characteristic curve can be used to schedule rope cutbacks and end reversals. This phase of the application will best be considered at a later date.

References

[1] U. S. Bureau of Mines: *Safe Practices in Mine Hoists*. Circular 61.

[2] R. M. Raymond: Safety Practice for Hoisting Ropes. Discussion, *Trans.* AIME (1923) **68**, pp. 182-188.

Phosphate Rock as an Economic Source of Fluorine

by W. L. Hill and K. D. Jacob

Fluorine recovery in the United States has been restricted chiefly to manufacture of ordinary superphosphate and wet-process phosphoric acid. However, there is an expanding use of fluorine by industry in the form of hydrofluoric acid. Improved methods for recovering fluorine from phosphate rock would yield an adequate supply of fluosilicates and a substantial contribution to the hydrofluoric acid requirement.

THE bulk of natural phosphates is comprised of calcium phosphates, which are usually apatites;[1] calcium aluminum phosphates such as pseudowavellite;[2] and aluminum phosphates, which occur in extensive deposits, notably on Grand Connetable Island.[3] Nearly all deposits of current commercial value are apatite, often with admixed material of other classes. In the trade coarsely crystalline material, mainly fluorapatite of igneous origin, moves as apatite, whereas the fine-grained material of sedimentary origin is marketed as phosphate rock. The latter classification includes the phosphate shales in western North America. Although several genetic varieties of phosphate rock are recognized,[4] most known reserves are of marine origin. Calcium phosphate is concentrated from Florida land pebble and Florida hard rock deposits and from Tennessee brown rock deposits.

Phosphate Rock as a Fluorine Carrier: More than two decades ago Reynolds and Jacob surveyed the fluorine content of commercial phosphate rock from various parts of the world.[3, 5] Later these samples were re-analyzed by a new and more reliable method of determining flourine.[6, 7] The revised results with interim additions of new samples, published a few years ago in a brief summary,[8] are shown in Figs. 1-3. Through the points representing the respective varieties of rock medial straight lines are drawn. In some instances the lines are well defined by the points; in others, where scattering renders the position of the best line uncertain, the greater weight is given points near the end of the covered range. Wherever distinct varieties are not recognized among regional deposits the custom is to identify the sample with the geographical location of the deposit.

Although all rock varieties cannot be classified with certainty, the plotted results exhibit three distinct types of rock in which the F to P_2O_5 ratio diminishes, remains sensibly constant, and increases, respectively, as the grade of rock increases. Existence of two types of Florida land pebble, Fig. 1, is supported by additional results recently supplied by a producer.[9]

Generally speaking, phosphate rock from continental deposits carries considerably more fluorine, and apatite and insular phosphates less, in proportion to the phosphorus content than is required by the formula for fluorapatite. Exceptions to this are apatite from Canada and certain samples of Florida waste-pond phosphate. The manner in which excess fluorine is held and the nature of the carbonate in carbonate apatite are discussed in recent articles.[10-12] High-grade calcium phosphates with F to P_2O_5 ratios less than 0.05 have been found in Curacao, Christmas Island,[8] Mexico,[13] and elsewhere.

The calcium aluminum phosphates and aluminum phosphates often carry notable amounts of fluorine. The F to P_2O_5 ratio for one sample of pseudowavellite concentrate was 0.05.[2] Aluminum phosphate from Grand Connetable Island is very low in fluorine, whereas some aluminum phosphate minerals[3] carry as much fluorine as highly fluorinated phosphate rock.

Fluorine in Domestic Phosphate: The curves shown in the figures permit interpolation of the F to P_2O_5 ratio corresponding to the midpoint of selected ranges in grade of any one of the several varieties of phosphate. Multiplication of the interpolated ratio by the mid-range grade (P_2O_5) yields a figure for the percentage of fluorine in this grade. Then, with the use of the mid-range grade, the amount of fluorine held in phosphate reserves can be estimated, see Table I. Accordingly, on Jan. 1, 1950, the fluorine complement of known domestic reserves exceeded 420,000,000 long tons of the element. A previous figure,[14] deduced in 1940 by a simpler procedure

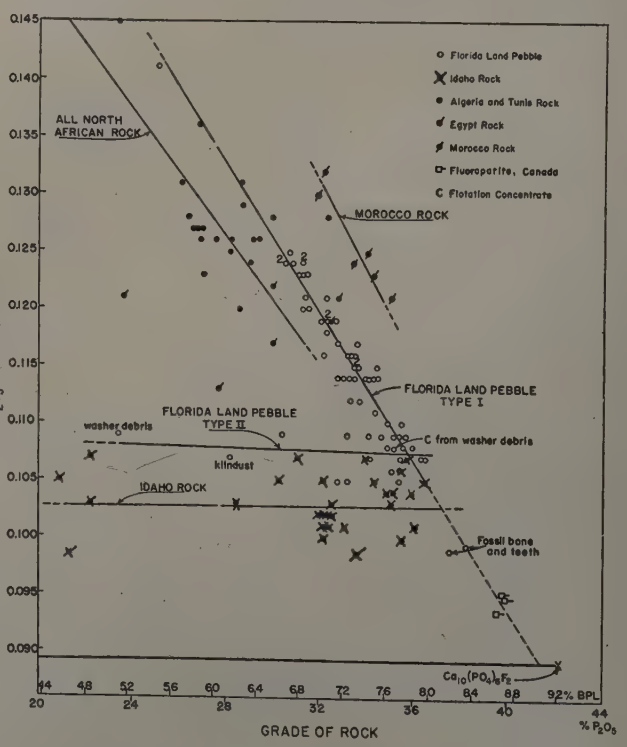


Fig. 1—The fluorine-phosphorus ratio in phosphate rock: Florida land pebble, rock from Idaho and North Africa, and apatite from Canada.

W. L. HILL and K. D. JACOB are Principal Chemist and Head, respectively, Division of Fertilizer and Agricultural Lime, Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, U. S. Dept. of Agriculture, Beltsville, Md.

Discussion on this paper, TP 3908H, may be sent (2 copies) to AIME before Dec. 31, 1954. Manuscript, Nov. 12, 1953. Nova Scotia Meeting AIME, September 1953.

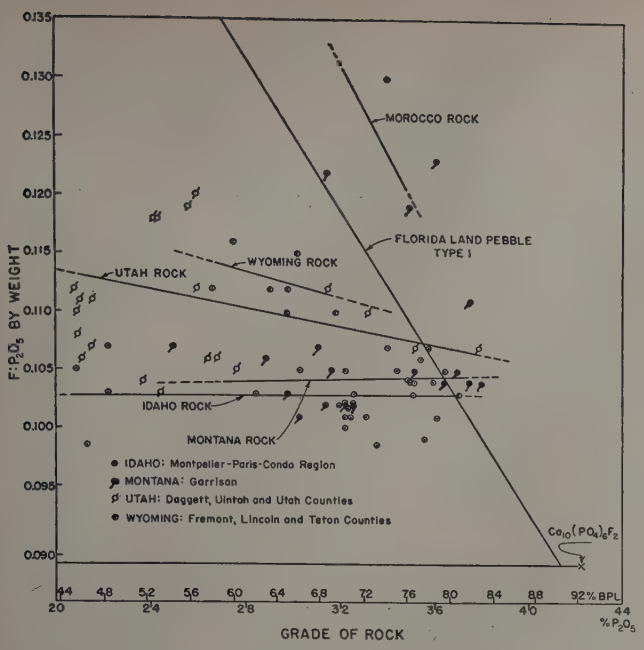


Fig. 2—The fluorine-phosphate ratio in phosphate rock from western United States.

for a reserve some 19 million tons below the 1950 estimate, is 415,192,000 long tons.

From published figures for domestic production of phosphate rock, and the tonnage of P_2O_5, it is possible to calculate the average grade and determine the F to P_2O_5 ratio by interpolation from the graphed results, see Table II. From this calculation the 1950 U. S. marketed production of phosphate rock carried 421,000 tons of fluorine.

Fluorine Carried in Rock Going to Manufacturing Processes: Marketed production of rock in 1950 was divided[15] among uses as follows: exports, 17 pct; direct application to soil and fertilizer filler, 9 pct; stock and poultry feed, 1 pct; chemical processing, 73 pct. By far the larger share of rock going to chemical processing was used in production of superphosphate, phosphoric acid, and phosphorus. It should be noted that electrothermal phosphoric acid,[16] see Table III, is now produced solely from phosphorus and that a very large part of the phosphorus production is thus consumed. Most, if not all, the production of Florida hard rock was used in phosphorus manufacture; the entire marketed production, see Table II, is assumed to have been so used. Indicated tonnages for other varieties listed under Phosphorus in Table III were deduced indirectly. The difference between the marketed production going to *superphosphates* and to *phosphates, phosphoric acid, phosphorus,* and *ferrophosphorus*[15] and that used in the manufacture of ordinary superphosphates, wet-process phosphoric acid, and triple superphosphate, Table III, is presumably the rock used in furnace processes of manufacture, that is, phosphorus (and electrothermal phosphoric acid) and other furnace processes. Tonnages thus obtained can be reconciled with the elemental phosphorus production, 153,233 short tons, on the basis of an apparent overall recovery of 80 pct of the rock phosphorus going to phosphorus manufacture and leave a balance of 118,350 tons of rock (P_2O_5 content indefinite) as the apparent consumption in the other furnace processes. In the computation the latter quantity of rock was divided equally between Florida land pebble and Tennessee rock, see Table III, and deducted from the respective total quantities indicated for furnace processes.

The recovery factor was derived from the 87.2 pct of the phosphorus actually charged into the furnace, found by tests[17] in which the slag carried 1.0 pct P_2O_5, which would reduce the stated factor to 83.6 pct. Allowance for a 1 pct P_2O_5 loss in preparation of the furnace feed and for 3 pct moisture in the rock reduces this factor to the figure employed.

Phosphate rock going to these major chemical processes in 1950 carried about 295,000 short tons of fluorine. Summation of percentages for Florida land pebble shows that 82 pct of the fluorine was carried in this variety of rock. Interestingly enough, the quantity of fluorine fed into these processes nearly equals domestic shipments of fluorspar of all grades, 301,510 short tons,[18] from the mines in 1950.

Fluorine Evolution: Results for the percentage evolution of fluorine in the several processes involved in domestic manufacture of superphosphate, phosphoric acid, and phosphorus are assembled in

Table I. Estimated Quantity of Fluorine in Known Domestic Phosphate Reserves as of Jan. 1, 1950

Variety or Location of Deposit	Phosphate Reserves,* Long Tons	Fluorine** Content, Pct	Fluorine in Reserves, Long Tons
Florida			
Land (and river) pebble	3,499,892,000	3.69†	129,146,015
Hard (and soft) rock	>1,539,778,000	2.99	> 46,039,362
Tennessee			
Brown (and white) rock	120,257,000	3.01	3,619,736
Blue rock	83,233,000	2.95	2,455,373
Western States			
Idaho and Montana	6,123,307,000	2.92	178,800,565
Utah and Wyoming	1,935,065,000	3.10	59,987,015
Other			
South Carolina land rock	8,798,000	3.12	274,498
Virginia apatite	2,000,000	2.85	57,000
Total	>13,312,330,000		>420,379,564‡

* Figures given by Jacob[89] for material containing 40 or more pct BPL.
** Medial percentages are given for varietal combinations.
† Result for main series (Type I).
‡ Rounded figure in short tons is 471,000,000.

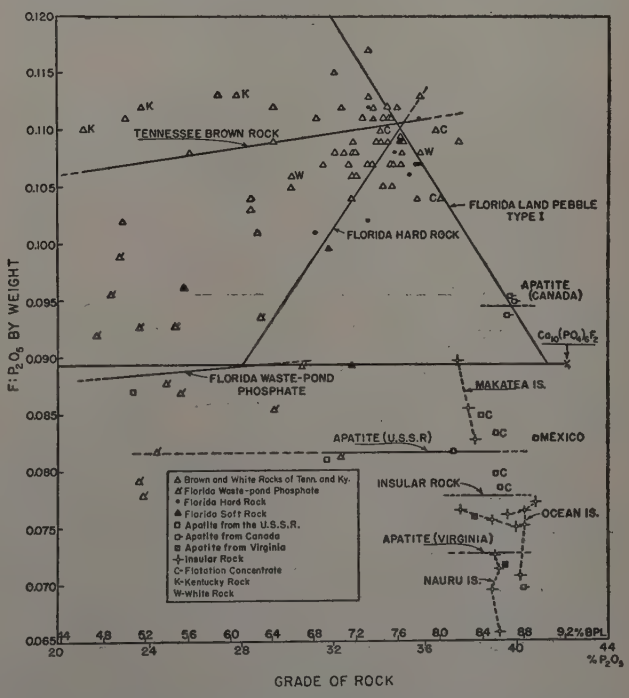


Fig. 3—The fluorine-phosphate ratio in phosphate rock: Florida hard-rock and waste-pond phosphates, Tennessee brown rock, various insular phosphates, and apatite from sundry deposits.

Table IV. Unfortunately, figures covering major rock varieties in all manufactures are not available, being notably deficient for phosphorus. Information of a qualitative nature from sundry sources indicates that fluorine evolution from Tennessee brown rock in smelter operations is not greatly different from that occurring from other varieties under the same conditions. Suitable combinations of the percentage

Table II. Fluorine in Marketed Production of Domestic Phosphate Rock in 1950

Variety and Source of Rock	Sold or Used by Producers* Rock, Long Tons	Sold or Used by Producers* P_2O_5, Long Tons	Average** P_2O_5 Pct	$F:P_2O_5$†	F, Long Tons
Florida					
Land pebble	7,933,009	2,673,882	33.7	0.114	304,823
Hard rock	71,319	25,133	35.4	0.111	2,790
Soft phosphate	81,542	16,994	20.8	0.0680	1,156
Western states					
Idaho, Utah and Wyoming	573,044	163,272	28.5	0.108	17,633
Montana	210,165	66,641	31.7	0.104	6,931
Tennessee					
Brown rock	1,384,473	390,190	28.2	0.109	42,531
Total	10,253,552	3,336,112			375,864‡

* Figures reported by Johnson and Jensen.[15]
** Calculated from figures in preceding columns.
† Obtained from Figs. 1, 2, and 3 for average grades using medial values of varietal combinations.
‡ Rounded figure in short tons is 421,000.

evolutions permit computation of the quantities evolved, as illustrated in Table V. Among tonnage data the figure for evolution in triple superphosphate production with wet-process acid is perhaps the least secure, because of the diversity in triple superphosphate manufacture. Plant to plant variations which influence the evolution markedly include acidulation with production acid, with moderately concentrated acid, and with concentrated acid. In the first two cases the product is kiln-dried, a process now sometimes merged with a granulation step, whereas with concentrated acid it is pile-cured. On the basis of published results[19] and information from private sources, it appears that about 42 pct of the production of wet-process acid was concentrated from an average production grade of 24.1 pct P_2O_5 to grades averaging 45.9 pct P_2O_5. The total quantity of phosphoric acid going to triple superphosphate production was divided as follows: wet-process acid 79.5 pct and electrothermal acid 20.5 pct.[19] The fluorine carried into the manufacture by the rock, 10,028 tons, is divided in this proportion between treatments by the two varieties of acid.

Other types of phosphate processing in which fluorine is given off include treatment of rock with gaseous P_2O_5 to produce calcium metaphosphate;[20] thermal defluorination of phosphate rock[21, 22] or a blend of rock and phosphoric acid;[23] calcination of rock with alkali salts to produce a Rhenania type of phosphate,[24, 25] or with magnesium silicates to produce calcium-magnesium phosphate silicate glass;[26, 27] defluorination of ordinary superphosphates;[28] granulation of ordinary superphosphate; and rock-drying operations. As a rule negligible evolution occurs in the latter treatment.

Defluorinated superphosphate, a secondary product prepared by heating superphosphate under conditions that volatilize 90 to 95 pct of the fluorine, is not included in the figures for defluorinated rock. Volatilization in the manufacture of calcium-magnesium phosphate-silicate glass ranges from 10 to 20 pct;[27] domestic production has ceased. Fluorine evolution in the manufacture of Rhenania-type phosphate is influenced markedly by conditions of processing. As the process is operated in Germany[25] for fertilizer manufacture with the use of apatite, for example, and a furnace temperature of 1100° to 1200°C, volatilization amounts to about 8 pct of the fluorine carried in the apatite.

In calcium metaphosphate production about 70 pct of the fluorine in the rock treated is evolved and the product contains about 63 pct P_2O_5 and 0.5 pct F.[20] Production in 1950 amounted to 15,500 tons of P_2O_5. According to these data the evolution must have been about 300 tons. Furthermore, published results[20] indicate that, tonnage-wise, the ratio of rock used to product obtained is near 0.535; with data already given this means that 13,000 tons of rock were treated. Deduction of this quantity of rock from the

Table III. Rock Used in Superphosphate, Phosphoric Acid, and Phosphorus Production in the United States in the Calendar Year 1950

Variety of Rock	Quantity, Short Tons	Grade Range, BPL, Pct	Fluorine Content,* Pct	Fluorine Quantity, Short Tons	Fluorine Share of Total, Pct
Ordinary Superphosphate**					
Florida land pebble	5,072,663	70 to 77	3.74	189,718	64.3
Tennessee brown rock	68,399	68 to 75	3.60	2,462	0.8
Rock from western U. S.	110,350	68 to 80	3.59	3,962	1.3
Total	5,251,412	68 to 80		196,142	66.4
Wet-Process Phosphoric Acid**					
Florida land pebble	885,424†	72 to 79	3.80	33,646	11.4
Tennessee brown, Idaho and Wyoming rocks	177,506	66 to 70	3.36	5,964	2.0
Total	1,062,930	66 to 79		39,610	13.4
Phosphorus (and Electrothermal Phosphoric Acid)					
Florida land pebble	274,669	60 to 68	3.75	10,300	3.5
Florida hard rock	79,877	‡	3.93	3,139	1.1
Rocks from Tennessee and western U.S.	1,362,295	§	2.62	35,692	12.1
Total	1,716,841			49,131	16.6
Triple Superphosphate (Phosphoric Acid Treatment of Rock)**					
Florida land pebble	225,993	72 to 76	3.73	8,430	2.9
Tennessee brown, Idaho and Wyoming rocks	48,129	64 to 70	3.32	1,598	0.6
Total	274,122	64 to 76		10,028	3.5
All varieties and processes	8,423,655	ca. 52 to 80		294,911	100.0

* Obtained from F to P_2O_5 ratios shown by graphed data for the grade range given in preceding column.
** Quantities and grades of rock going to this manufacture are given by Adams et al.[19]
† Inclusive of 11,539 tons of 68 pct BPL rock, which has been neglected in the grade computations, being a small fraction of the varietal total going to this use.
‡ Only the average grade is known (Table II).
§ The average grade of rock going to phosphorus manufacture in the western phosphate field is said to be 24 pct at the present time.

Table IV. Fluorine Volatilization in Domestic Manufacture of Superphosphate, Phosphoric Acid, and Phosphorus

Rock Variety, Product, or Treatment	Fraction of Rock Fluorine Evolved			
	Determinations, Number	Range, Pct	Average, Pct	Mid-range, Pct
Ordinary Superphosphate*				
Acidulation step:				
Florida land pebble	19	16.5 to 37.8**	26.4	27.1
Tennessee brown rock	11	28.1 to 42.1	34.1	35.1
Idaho and Montana rock	3	33.5 to 38.3	35.7	35.9
All four varieties (30 factories)	33	16.5 to 42.1	29.9	29.3
Granulation step:				
Florida land pebble (experimental runs)	3	0.6 to 2.2	1.7	1.4
Wet-Process Phosphoric Acid*				
Production step (acid, 20 to 30 pct P_2O_5):				
Florida land pebble (sodium phosphate industry)				≦1.0
All four varieties (fertilizer industry)	4	5.2 to 23.7	13.0	14.5
All four varieties (both industries)		<1.0 to 23.7		12.0
Concentration step (acid, 39 to 50 pct P_2O_5):				
All four varieties (concentration step only)	4	10.0 to 38.7	27.4	24.4
Phosphorus (and Electrothermal Phosphoric Acid)*				
Preliminary treatment of rock:				
Tennessee brown rock, nodulization		12.8 to 40		26.4
Tennessee brown rock, sintering		10 to 20		15.0
Tennessee brown rock, briquetting and calcination		10 to 20		15.0
Tennessee brown rock, all treatments		10 to 40		25.0
Smelting process:				
Pre-treated rock		0.3 to 4†		2.2†
Raw lump rock		20 to 30		25.0
Triple Superphosphate (Phosphoric Acid Treatment of Rock)*				
Acidulation step:				
Wet-process acid, 41.7 to 44.7 pct P_2O_5	2	7.5 to 13.4‡§		10.5§
Electrothermal acid, 54 to 56 pct P_2O_5			4	
Acidulation and kiln-drying steps:				
Wet-process acid, 39.0 to 41.7 pct P_2O_5	2	13.6 to 50.2§§		31.9§

* Results given by Jacob et al.[38] Supplemental data for wet process phosphoric acid were supplied privately by Blockson Chemical Co. Supplemental data for phosphorus (and electrothermal phosphoric acid) were supplied privately by Monsanto Chemical Co., Tennessee Valley Authority, and Victor Chemical Works.

** Result for Oberphos and one obviously unreliable result given in the reference article were disregarded in determination of the range.

† Divided, perhaps about equally,[38] between dust and gaseous fluorine compounds; result calculated on supposition that 90 pct of fluorine in smelter furnace feed remains in the slag.[17,38]

‡ Results for Idaho rock (acid, 44.7 pct P_2O_5) and Tennessee brown rock (acid, 41.7 pct P_2O_5), respectively.

§ Percentage of fluorine carried into process by both rock and acid.

§§ Results for Montana rock (acid, 39.0 pct P_2O_5) and Tennessee brown rock, respectively.

118,350 tons assigned to thermal processes other than phosphorus (and electrothermal phosphoric acid) manufacture yields the figure 105,350 tons, which may be regarded as the apparent rock consumption in production of defluorinated rock. With the usual volatilization of 90 to 98 pct attained in defluorination processes this quantity of rock would yield 3000 to 4000 tons of evolved fluorine.

Division of Fluorine Among Products: In ordinary superphosphate manufacture the fluorine divides between the product and surrounding atmosphere. Evolution is restricted almost entirely to mixing and denning operations. Only negligible amounts escape from the curing pile. On the other hand, in the manufacture of phosphoric acid and triple superphosphate the fluorine, dividing at three or more process stages, is distributed among several products. A possible distribution among the products obtained in the manufacture of wet-process phosphoric acid, triple superphosphate, and sodium phosphates is illustrated in Fig. 4. It must be realized that although individual results all lie within ranges found in industrial practice, the chart does not depict any actual integrated sequence of steps. The division of fluorine shown for the production-acid step was deduced from results from fertilizer operations. By a suitable choice of conditions the quantity going into the filter cake, as well as that evolved, can be made very small. Apparently such practice is followed by some manufacturers of sodium phosphate, with the result that nearly all the fluorine goes into the production acid and is thereafter collected by precipitation from solution.

A probable division of fluorine among products obtained in manufacture of electrothermal phosphoric acid is illustrated in Fig. 5. The fluorine content of condensed phosphorus runs 10 ppm or less when the condenser is operating properly. The fluorine is held in occluded condenser water.

Disposal of Evolved Fluorine: Nearly 25 years ago it was observed[5] that whereas superphosphate manufacturers usually absorbed evolved fluorine in water as a disposal measure, a few big producers recovered a large part in the form of marketable fluosilicates, whereas small producers merely ran

Table V. Estimated Fluorine Evolution in Domestic Production of Superphosphate, Phosphoric Acid, and Phosphorus in Calendar Year 1950

Manufacture	Fluorine Entering Process,* Pct	Fluorine Evolved		
		Fraction** of Total, Pct	Quantity, Short Tons	Share in Total Evolution, Pct
Ordinary superphosphate (acidulation step)	196,142	29	56,881	69.5
Wet-process phosphoric acid:				
Production step	39,610	12	4,753	5.8
Concentration step	16,636	24	3,993	4.9
Phosphorus (and electrothermal phosphoric acid)	49,131	26†	12,774	15.6
Triple superphosphate:				
Wet-process acid	16,000‡	21§	3,360	4.1
Electrothermal acid	2,056	4	82	0.1
Total	294,911		81,843	100.0

* Unless it is otherwise indicated in a footnote, the figures are the section totals given in Table III.

** Unless it is otherwise indicated in a footnote, figures are the mid-range results (rounded) shown in Table IV.

† Rounded average of overall figures for evolution with and without pre-treatment (Table IV, Phosphorus and Electrothermal Phosphoric Acid).

‡ Figure includes 7972 tons carried in the rock and an estimated figure for that in the acid.

§ Rounded average of figures for acidulation step and acidulation step with kiln drying (Table IV, Triple Superphosphate).

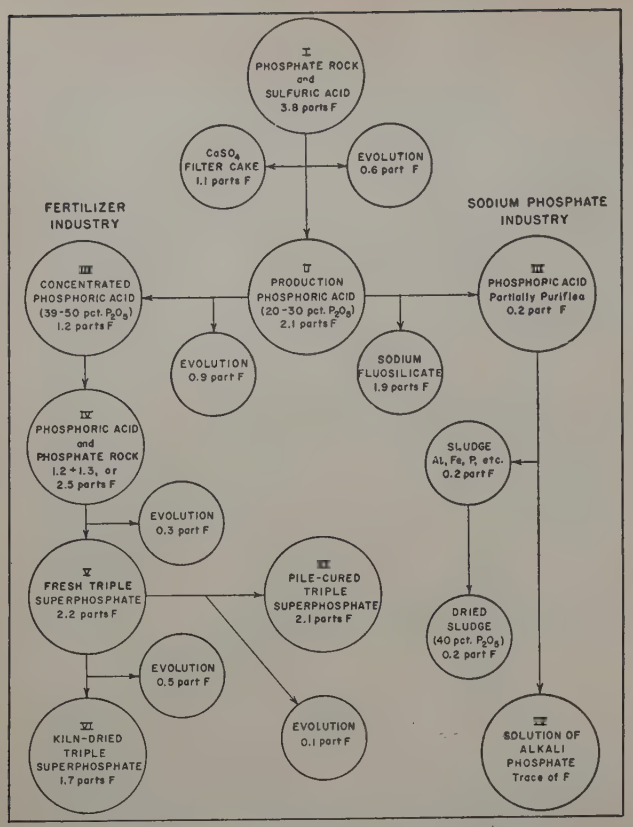

I
PHOSPHATE ROCK and SULFURIC ACID 3.8 parts F
CaSO4 FILTER CAKE 1.1 parts F
EVOLUTION 0.6 part F
FERTILIZER INDUSTRY
SODIUM PHOSPHATE INDUSTRY
III
CONCENTRATED PHOSPHORIC ACID (39-50 pct. P2O5) 1.2 parts F
V
PRODUCTION PHOSPHORIC ACID (20-30 pct. P2O5) 2.1 parts F
III
PHOSPHORIC ACID Partially Purified 0.2 part F
EVOLUTION 0.9 part F
SODIUM FLUOSILICATE 1.9 parts F
IV
PHOSPHORIC ACID and PHOSPHATE ROCK 1.2 + 1.3, or 2.5 parts F
SLUDGE Al, Fe, P, etc. 0.2 part F
EVOLUTION 0.3 part F
V
FRESH TRIPLE SUPERPHOSPHATE 2.2 parts F
III
PILE-CURED TRIPLE SUPERPHOSPHATE 2.1 parts F
DRIED SLUDGE (40 pct. P2O5) 0.2 part F
EVOLUTION 0.5 part F
VI
KILN-DRIED TRIPLE SUPERPHOSPHATE 1.7 parts F
EVOLUTION 0.1 part F
III
SOLUTION OF ALKALI PHOSPHATE Trace of F

Fig. 4—Illustration of likely division of fluorine in manufacture of wet-process phosphoric acid, triple superphosphate, and sodium phosphate. (Based on data in Table IV, results given by Jacob et al,[38] and unpublished results).

the absorbed fluorine compounds to waste. At present the bulk of the fluorine evolved in manufacture of phosphoric acid, superphosphate, and other phosphorus-bearing products is removed from the exit gases before they are discharged to the atmosphere. The fluorine is usually either scrubbed out with water or absorbed in a bed of suitable solid materials. Fluorine-bearing solution from the scrubber is often neutralized with hydrated lime before it is run to waste. In certain thermal processes fluorine in the stack gas is rendered innocuous by introduction of lime dust at the base of the stack.

Recovery of Fluorine in Marketable Form: The ideal solution to disposing of evolved fluorine would be to recover it in the form of useful compounds. Unfortunately, at the present time the cost of recovery would often exceed the market value of the product. The quantity of fluorine recovered in phosphate processing is indicated to a fair degree of reliability by the production of fluosilicic acid and the fluosilicates of potassium, ammonium, magnesium, zinc and, principally, sodium. Total output of these materials in 1950 was equivalent to 13,970 short tons of fluorine. This is slightly under 17 pct of the total indicated evolution, see Table V, or about 20 pct of the evolution in manufacture of superphosphate and wet-process phosphoric acid. However, this figure representing total recovery includes non-evolved fluorine reclaimed in the sodium phosphate industry. Actual recovery of volatilized fluorine was less than one-sixth of the indicated total evolution.

The low score on recovery of evolved fluorine can be attributed to unfavorable distribution of evolution and to lack of incentive for recovery. Comparison of results in Tables V and VI reveals points in the process where recovery is most promising. Sizable rock tonnage and high percentage of volatilization present a very favorable combination, and ordinary superphosphate stands high in this regard. Production, however, is widely scattered, see Table VI. The 1951 production came from 204 plants in 32 states. Furthermore, 53 pct came from 156 plants in which average productions were less than 50,000 tons and required treatment of less than 25,000 tons of rock. Since the average fluorine content of all rock used for manufacture of superphosphate is 3.73 pct, the fluorine complement of this quantity of rock is only 930 tons, of which 29 pct, see Table V, or 270 tons is estimated evolution. In the light of these figures the paucity of recovery units in proportion to the number of plants is understandable. According to a recent survey[19] only 19 recovery units were coexistent with ordinary superphosphate plants in 1951. Five of these were not being operated, probably because a weak fluosilicate market did not offer sufficient incentive.

Table VI. Ordinary Superphosphate Production in Relation to Size of Plants for the Calendar Year 1951*

Annual Capacity, Thousand Short Tons	Number of Plants**	Average Plant Capacity Short Tons	Production	
			Average Plant Production, Short Tons	Group Share in Total, Pct
<25	7	17,600	9,800	0.7
25 to 50	35	40,000	20,100	7.2
50 to 75	60	61,900	34,300	20.9
75 to 100	54	85,900	44,600	24.5
100 to 200	42	123,300	74,200	31.7
>200	6	261,800	245,900	15.0

* The figures, representing production in Continental United States in terms of superphosphate containing 18 pct of available P_2O_5, are adaptations of data given by Adams et al.[19]
** This summary covers 204 plants scattered over 32 states; two of the plants discontinued operation during the year.

Similar conditions prevail in the manufacture of wet-process phosphoric acid and triple superphosphate. In 1951 13 plants producing wet-process acid shared in the rock equivalent of nearly 40,000 tons of fluorine, see Table III, with an average of 3000 tons each. Only two plants had recovery facilities. Of the nine plants producing triple superphosphate only one recovered fluorine.

Fluorine evolved in the phosphorus smelter furnace is caught in the phosphorus condenser water. Small amounts of fluosilicates were recovered from this source in England for a number of years prior to 1914.[29] As far as the authors are aware recovery from this source has not been practiced elsewhere. Recent work on recovery of fluorine evolved in a defluorination plant[21] points to a promising method that should be generally applicable to furnace stack gases. The process, put into regular operation a short time ago, produces a metallurgical grade of calcium fluoride.

Trends and Outlook

It remains to list and evaluate changes in the fluorine economy that are now under way or may come in the future. Attention is directed to actual and possible changes in patterns of evolution, recovery and markets, and needed technological advances.

Evolution: Undoubtedly fluorine evolution has increased since 1950 because larger tonnages of rock have been going to chemical processes. In comparison with 1950 percentage production increases for the chief primary products in 1951 and 1952 are, respectively: ordinary superphosphate, 2 and 5; wet-process phosphoric acid, 17 and 34; phosphorus (and electrothermal phosphoric acid), 12 in 1951; triple superphosphate, 7 and 26. If the pattern of evolution had not changed during the interim, the

1952 evolution would have been 10 pct above that for 1950. Probably the increase was appreciably less than 10 pct, because steadily increasing quantities of wet-process phosphoric acid are being utilized without concentration in the manufacture of fertilizer-grade ammonium phosphate and feed-grade dicalcium phosphate. Nitric acid treatment of phosphate rock, about ready for commercial development for production of fertilizers, promises little for fluorine evolution in phosphate processing.

In the future, procedures may retain fluorine at unfavorable points in the process for expulsion at more favorable places. Also, business developments may concentrate production in a few large operations. The expanding triple superphosphate industry has exhibited a trend toward production concentration, although preparation of this material in the conventional ordinary superphosphate plant is a step in the other direction.

Recovery: Fluorine recovery in phosphate processing in the United States has been restricted mainly to manufacture of ordinary superphosphate and wet-process phosphoric acid, with some contribution from triple superphosphate. The products are fluosilicic acid and its salts. As indicated earlier in this article, it is now being recovered from furnace stack gas in the form of a metallurgical grade of calcium fluoride. The one recovery unit now in operation undoubtedly will demonstrate the possibilities of recovery from furnace stack gas. It is to be hoped that this will lead to improvement in the quality of calcium fluoride and encourage installations for recovery from this source. The winning of marketable fluorine compounds from phosphate solutions, heretofore practiced mainly in the sodium phosphate industry, is feasible in the manufacture of feed-grade dicalcium phosphate from wet-process acid. Recovery in this manner is anticipated in the new plants for this manufacture. Although fluorine evolution would be affected adversely by increased production of dicalcium phosphate for feed use, since concentration of the phosphoric acid is not necessary, recovery would probably be enhanced as a consequence of the precipitation technique.

Market Pattern: The fluosilicate market was satisfied in 1950 by fluorine recovery in phosphate processing without the utilization of the full recovery capacity. At the same time a part of the fluosilicate was consumed in the manufacture of sodium fluoride. It can be concluded, therefore, that phosphate rock was at that time an economic source of some 14,000 short tons of fluorine, which was supplied mainly by large operations. Recently the advent of water fluoridation has increased the demand for sodium fluosilicate and thus greatly stimulated interest in fluorine recovery. The significance of this new use as a factor in the fluosilicate market can be made obvious with the aid of a few figures. In 1950 13,600 million gallons of water per day were furnished by public water supplies in the United States.[30] An unpublished estimate for 1952 is 16,000 million gallons per day. For fluoridation 1 part F per million parts water is the addition generally recommended,[31] and either the fluoride or fluosilicate of sodium can be used. Accordingly 21,000 short tons of fluorine would have been required to fluoridate the entire public supply in 1950; by 1952 an additional 3000 tons would have been needed. It is estimated[32] that 100 million people are served by public supplies, of which 3.5 million reside in areas where adequate fluorine is carried in the water. Thus 96.5 pct of 24,000, or 23,200 tons, would have been the limiting tonnage for this use in 1952. On July 15, 1953, fluoridated water was used by 14.5 million people, about twice the number in the preceding year, whereas other communities representing 15.6 million people are now considering fluoridation. Supposing that the proposed adoptions materialize promptly, the indicated growth of fluoridation is 7, 15, and 30 pct of the public water supply in the years 1952, 1953, and 1954, respectively. Combination of the foregoing results gives the probable tonnages of fluorine required for this use: 1700 in 1952, 3800 in 1953, and 8100 in 1954. Obviously before very long fluoridation will be taking a quantity of fluorine somewhat greater than one-half of the 1950 recovery from phosphate processing. Problems connected with fluoridation in a large plant are discussed in a recent article.[33]

There is an expanding use of fluorine by industry in the form of hydrofluoric acid.[34–36] Industrial consumption increased from 15,500 tons of hydrofluoric acid[33] in 1940 to 36,500 tons[34] in 1950, and the estimates for 1951 and 1952[37] are 44,000 and 47,500 tons. Its principal uses are in aluminum metallurgy, petroleum alkylation, and the manufacture of refrigerants, aerosol propellants, and polyterpene resins. The supply of this chemical comes mainly from fluorspar. In 1950 acid-grade fluorspar shipments[18] from domestic mines carried about 46,000 tons of fluorine. The acid-grade material commands a premium price. Prior to 1952 the known accessible reserves were alarmingly inadequate to meet growing demand, although the lower grades were in plentiful supply. The gap has now been closed, and ample supply of acid-grade material is in sight.

Needed Technological Development: At the present time phosphate rock going to chemical processing carries several-fold the quantity of fluorine required by the market demand for fluosilicates and hydrofluoric acid. The indicated actual evolution in 1950 exceeded market demand by about 13 pct. This source could provide not only an adequate supply of fluosilicates but also a substantial contribution to the hydrofluoric acid requirement. Achievement of these

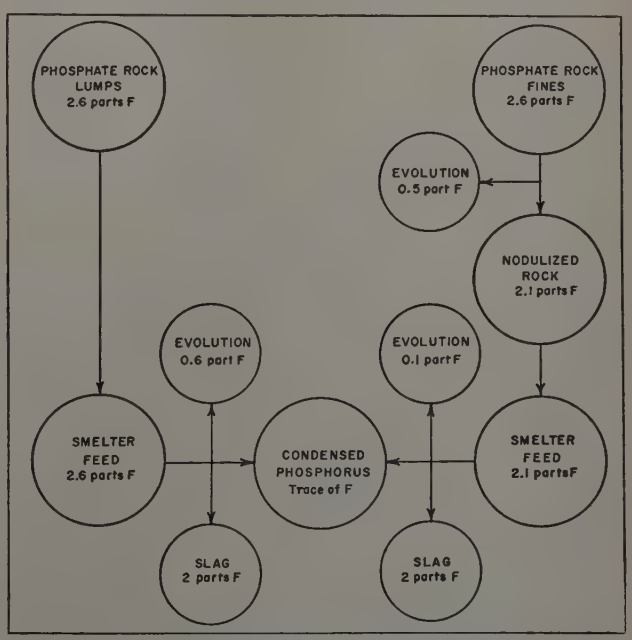

PHOSPHATE ROCK LUMPS 2.6 parts F
PHOSPHATE ROCK FINES 2.6 parts F
EVOLUTION 0.5 part F
NODULIZED ROCK 2.1 parts F
EVOLUTION 0.6 part F
EVOLUTION 0.1 part F
SMELTER FEED 2.6 parts F
CONDENSED PHOSPHORUS Trace of F
SMELTER FEED 2.1 parts F
SLAG 2 parts F
SLAG 2 parts F

Fig. 5—Illustration of likely divisions of fluorine in manufacture of electrothermal phosphoric acid. (Based mainly on data given in Table IV).

objectives depends on improvements in current recovery practice.

There is need for improved recovery methods to lower equipment cost and/or increase percentage recovery of fluorine evolved in the manufacture of superphosphate and wet-process phosphoric acid. It is said that some recovery units do not reclaim more than 50 pct of the localized evolution. A desirable development for the small plant would be a midget recovery unit.

A method for producing acid-grade calcium fluoride from furnace gases is also needed. This might be an improvement in the TVS process,[21] which now can produce only the lower grades. A greater economic opportunity would be offered by a method for producing hydrofluoric acid directly from furnace gases, since the acid has a much higher market value. Still another way to obtain hydrofluoric acid from phosphate rock used in fertilizer manufacture lies in the development of a process for economic production from fluosilicates. Until this process becomes a reality, the only hope for supplementing the hydrofluoric acid supply from phosphate rock processing is recovery of fluorine from furnace gas.

References

[1] C. Palache, H. Berman, and C. Frondell: *Dana's System of Mineralogy,* Vol. II, 7th ed. New York, 1951.

[2] W. L. Hill, W. H. Armiger, and S. D. Gooch: Some Properties of Pseudowavellite from Florida. *Trans.* AIME. (1950) **187**, p. 699.

[3] K. D. Jacob, W. L. Hill, H. L. Marshall, and D. S. Reynolds: The Composition and Distribution of Phosphate Rock with Special Reference to the United States. U. S. Dept. Agr. *Tech. Bull.* 364 (1933) 90 pp.

[4] E. Blackwelder: Origin of the Rocky Mountain Phosphate Deposits (Abstract). *Bull.* 26, Geol. Soc. Amer. (1915) p. 100.

[5] K. D. Jacob and D. S. Reynolds: The Fluorine Content of Phosphate Rock. *Journal of the Association of Official Agricultural Chemists* (1929) **11**, p. 237.

[6] D. S. Reynolds and W. L. Hill: Determination of Fluorine with Special Reference to Analysis of Natural Phosphates and Phosphatic Fertilizers. *Industrial and Engineering Chemistry* (1939) **11**, p. 21.

[7] H. H. Willard and O. B. Winter: Volumetric Method for Determination of Fluorine. *Industrial and Engineering Chemistry,* Anal. Ed. (1933) **5**, p. 7.

[8] K. D. Jacob: Phosphorus Supplements for Livestock—Sources, Supplies, and Fluorine-Phosphorus Relations. *Feedstuffs* (1944) **16**, No. 7, 18.

[9] W. T. Whitney: Private communication to W. L. Hill (1953).

[10] S. B. Hendricks and W. L. Hill: The Nature of Bone and Phosphate Rock. *Proc.,* National Academy of Science (1950) **36**, p. 731.

[11] Z. S. Altschuler, E. A. Cisney, and I. H. Barlow: X-ray Evidence of Nature of Carbonate-Apatite (Abstract). *Bull.* 63, Geol. Soc. Amer. (1952) p. 1230.

[12] D. McConnell: Viséite, a Zeolite with the Analcime Structure and Containing Linked SiO_4, PO_4 and H_xO_4 Groups. *American Mineralogist* (1952) **37**, p. 609.

[13] J. G. Cady, W. L. Hill, E. V. Miller, and R. M. Magness: Occurrence of Beta Tricalcium Phosphate in Northern Mexico. *American Mineralogist* (1952) **37**, p. 180.

[14] K. D. Jacob: New and Old Methods of Processing Phosphates. *American Fertilizer* (1940) **93**, No. 8, p. 7; No. 9, p. 7.

[15] B. L. Johnson and N. C. Jensen: Phosphate Rock. U. S. Dept. Interior, *Bureau of Mines Minerals Yearbook* (1950) p. 1003.

[16] M. M. Striplin, Jr.: Development of Processes and Equipment for Production of Phosphoric Acid. Tennessee Valley Authority *Chemical Engineering Report* No. 2 (1948) 143 pp.

[17] R. R. Burt and J. C. Barber: Production of Elemental Phosphorus by the Electric-Furnace Method. Tennessee Valley Authority *Chemical Engineering Report* No. 3 (1952) 312 pp.

[18] H. W. Davis: Fluorspar and Cryolite. U. S. Dept. Interior *Bureau of Mines Minerals Yearbook* (1950) p. 521.

[19] J. R. Adams, T. H. Tremearne, K. D. Jacob, and L. G. Porter: Survey of the Superphosphate and Wet-Process Phosphoric Acid Industries in the United States in 1950 and 1951. U. S. Dept. Agr., Bur. Plant Industry, Soils, and Agr. Eng. and Office of Materials and Facilities, Processed Report (1952) 44 pp.; New Data on Fertilizer Phosphates (editor's summary). *Chemical Engineering* (1952) **59**, No. 8, p. 142.

[20] J. C. Brosheer: Development of Processes for Production of Calcium Metaphosphate Fertilizer. Tennessee Valley Authority *Chemical Engineering Report* No. 6 (1953) 102 pp.

[21] J. C. Brosheer and T. P. Hignett: Development of Processes for Production of Fused Tricalcium Phosphate. Tennessee Valley Authority *Chemical Engineering Report* No. 7 (1953) 143 pp.

[22] W. T. Whitney and C. A. Hollingsworth: Production of Defluorinated Phosphate Rock—Calcining without Fusion in Rotary Kilns. *Industrial and Engineering Chemistry* (1949) **41**, p. 1325.

[23] C. A. Butt: Manufacture of Defluorinated Tricalcium Phosphate. U. S. Patent 2,442,969 (June 1948); 2,565,351 (Aug. 1951).

[24] K. D. Jacob: German Fertilizers and Soil Fertility. Official Military Government for Germany (U. S.), FIAT Final Report 665 (1945) pp. 32-35; U. S. Dept. Commerce, Office of Technical Services PB-18777.

[25] J. R. Hawes and F. M. Lea: Kali Chemie Rhenania Phosphat Werke Brunsbüttelkoog. U. S. Dept. Commerce, Office of Technical Services PB No. 18913.

[26] W. L. Hill, F. N. Ward, W. H. Armiger, and K. D. Jacob: Composition and Fertilizer Value of Phosphate Rock—Magnesium Silicate Glasses. *Journal of the Association of Official Agricultural Chemists* (1948) **31**, p. 381.

[27] R. W. Moulton: Electric Furnace Fertilizer—Ca-Mg-Phosphate. *Chemical Engineering* (1949) **56**, No. 7, p. 102.

[28] W. H. Waggaman: Phosphoric Acid, Phosphates and Phosphatic Fertilizers. A C S Monograph No. 34, 2nd ed. New York, 1952.

[29] R. E. Threlfal: The Story of 100 Years of Phosphorus Making 1851-1951. Albright and Wilson, Ltd., Birmingham, England, 1952.

[30] K. A. MacKichan: Estimated Use of Water in the United States—1950. U. S. Dept. Interior, Geol. Survey *Circ. 115* (1951) 13 pp.

[31] F. J. Maier: Fluoridation of Public Water Supplies. *Journal of the American Water Works Association* (1951) **43**, p. 1120.

[32] T. L. Hagan, U. S. Public Health Service: Private Communication.

[33] N. E. Jackson and E. A. Schmitt: The Washington Story of Fluoridation. *Water and Sewage Works* (1952) **99**, p. 435.

[34] T. J. Brice, H. G. Bryce, and H. M. Scholberg: Fluorochemicals—Today and Tomorrow. *Chemical Engineering News* (1953) **31**, p. 510.

[35] J. R. Callaham: Fluorine Industry Molds a Postwar Career from Wartime Service. *Chemical and Metallurgical Engineering* (1945) **52**, p. 94.

[36] E. M. Ott: Fluorine. *Chemical Engineering News* (1953) **31**, p. 1626.

[37] The Chememtator: Not Enough Hydrofluoric. *Chemical Engineering* (1952) **59**, p. 112.

[38] K. D. Jacob, H. L. Marshall, D. S. Reynolds, and T. H. Tremearne: Composition and Properties of Superphosphate—Volatilization of Fluorine in Superphosphate Manufacture. *Industrial and Engineering Chemistry* (1942) **34**, p. 722.

[39] K. D. Jacob: *Fertilizer Technology and Resources in the United States,* Agronomy Monograph, Vol. III, Chap. 5. New York, 1953.

Analysis of Variables In Rod Milling

by Will Mitchell, Jr., C. L. Sollenberger, T. G. Kirkland, and B. H. Bergstrom

SEVERAL constructive and fundamental studies have been made in the analysis of data obtained from experiments carried on with batch ball and rod mills. The operating characteristics of ball milling in small continuous circuits have also been appraised. It is from these analyses that some of the theories of comminution have been developed.

Relatively few studies of continuous rod milling have added significantly to the fundamental concepts, because seldom have they yielded sufficiently consistent results. Perhaps they have been too limited in their scope. Careful control of the variables in batch grinding is simple compared with that encountered in a continuous operation. This factor alone has discouraged many investigators. Occasionally results of systematic changes made in industrial rod mill circuits have been published, but usually the data are sketchy and are restricted because of the unwieldiness of the equipment used. The work, in general, has not been comprehensive; nevertheless it has provided empirical relationships that have bridged the gap between postulate and practice so that by proper manipulation of formulae, a mill designer can anticipate mill size and power requirements.[1-3] Although operating variables of a small continuous mill are not so easy to control as with the batch mill, with present day devices, and with careful experimental work, consistent results can be obtained.

Nearly four years ago, in the Process Laboratory, Allis-Chalmers Mfg. Co. began a systematic study of the effects of several variables upon the performance of the pilot rod mill. A mill was built in the laboratory to provide the versatility required for the proposed study. It was constructed in sections so that it could be operated, with a few modifications, as a rod mill 30 in. x 8 ft or 30 in. x 4 ft. The discharge end of the shell was flanged so that either an end peripheral discharge or an overflow discharge could be installed. Thus the performance of at least four types of mills could be studied merely by changing the type of discharge or the length of the mill shell.

The grinding experiments were designed so that a study could be made of the way in which the mill speed, feed rate, and pulp density influenced the performance of both overflow and end peripheral discharge rod mills. Four sets of experimental data were collected from the four mill arrangements. The mill in each set of experiments was fed at four rates of feed depending on the length of the mill, at four pulp densities, and at five percentages of critical speed. Electrical and mechanical controls were installed to regulate all these independent variables, and auxiliary devices were used to verify the precision of the controls at each point. The dependent variables used to quantify the experiments were the reduction ratio and the new surface area produced as calculated from sieve analyses. These were incorporated with the energy factor by the calculation of both the new surface produced per unit of energy and the Bond work index.[4] Rod wear, as a dependent variable, was not studied because of the short period of operation for each run.

W. MITCHELL, JR., C. L. SOLLENBERGER, T. G. KIRKLAND, and B. H. BERGSTROM, Members AIME, are with the Research Laboratories, Allis-Chalmers Mfg. Co., Milwaukee.

Discussion on this paper, TP 3901B, may be sent (2 copies) to AIME before Dec. 31, 1954 . Manuscript, July 7, 1954. New York Meeting, February 1954.

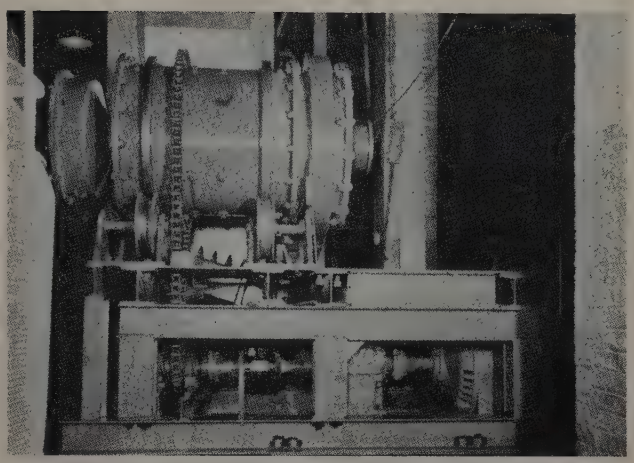


Fig. 1—A view of the 30-in. x 4-ft rod mill.

Exclusive of repeat runs, each set of experiments yielded 80 products, and the total study at least 320 products, all of which were quantified. With the operating information collected, these data presented a bewildering accumulation. Statistical analysis has been invaluable in unraveling the confusion and in presenting a means of establishing the nature and the magnitude of the significant variables.

Data presented in this paper are those from the 30 in. x 4 ft end peripheral discharge rod mill, Fig. 1, when limestone was ground at feed rates of 1000, 2000, 4000, and 5000 lb per hr, at pulp densities of 50, 60, 70, and 80 pct solids, and at mill speeds of 50, 60, 70, 80, and 90 pct of the critical speed. These 80 tests have all been run at least twice, and occasionally a third time, to prove that the data obtained were reproducible. The techniques of operation and the methods of quantification of results are described in the following pages and the results analyzed statistically to show the significant variables. The variables are plotted to show the relationships that exist.

A massive dolomitic limestone from Waukesha Lime and Stone Co. was used for feed during these experiments because of its availability and its textural uniformity. This limestone analyzed 28.7 pct CaO, 21.0 pct MgO, 6.0 pct SiO_2, 0.4 pct Al_2O_3, and 0.3 pct Fe_2O_3 and had a loss on ignition of 44.1 pct. It had a rod mill grindability at 14 mesh of 9.6 grams per revolution from which a work index of 13.9 was calculated. The ball mill grindability at

100 mesh was 3.34 gpr, which is equivalent to a work index of 7.28. Work indices calculated from Bond grindabilities through all the mesh sizes from 14 mesh to and including 200 mesh showed that the work index decreased consistently as the grind became finer. Apparently this limestone is easier to grind in the finer sizes.

Preliminary tests showed that a −¾ in. crushed stone as it was received from the supplier did not give a consistent sieve analysis; hence, a coarsely crushed stone was obtained from the quarry and a uniformly sized rod mill feed was prepared in the laboratory, see Fig. 2. Minus 1½-in. +¾-in. stone was received in 8-ton dump trucks and passed through a 2-in. grizzly onto a 20-in. conveyor belt. From the belt the stone was lifted by a bucket elevator and discharged into two 15-ton bins.

The bins were of the slanting-bottom type but had a horizontal square steel distribution plate located 3 ft above the discharge opening and 1 ft in from the four sides. This device allowed the material to flow from four directions into the bin discharge opening and eliminated the funneling through of feed from the vertical side. Sampling of the discharge from the bins showed no significant segregation, especially when the bins were maintained nearly full.

From the bins the stone was fed by means of electro-magnetic vibrating feeders to a Hydrocone crusher equipped with a 1½-in. eccentric and set at 0.4 in. close side. To check the crusher setting three lead slugs were passed through the chamber at least once an hour during the operating cycle. The slugs were collected from the discharge of the crusher and their thickness measured with a micrometer. When necessary, the crusher setting was adjusted.

The crusher produced a rod mill feed, 96.8 pct passing ¾ in., which was transferred by conveyor belt and bucket elevator into a third 15-ton bin similar to the ones described. Near the top and in the center of the rod mill feed bin, a Tellevel bin indicator was installed. Whenever the level of the stone settled below a certain point the indicator automatically activated the crushing circuit to produce more crushed stone until the bin was filled again. To minimize segregation, the rod mill feed bin was also equipped with a square steel distribution plate near the bottom.

An electromagnetic feeder at the discharge opening of the bin conveyed feed into the hopper of a No. WL-2669 Merrick Feedoweight. A Tellevel indicator in the hopper activated the feeder at the discharge of the feed bin whenever the level of the feed in the hopper dropped sufficiently. The Feedoweight maintained a constant feed rate within 50 lb for any given setting. It was also equipped with a *totalizer* which measured the total amount of feed traveling on the feeder belt for any interval of time. The Feedoweight discharged into a curved spout which discharged into a drum feeder on the rod mill.

The product from the mill flowed down a launder to a spiral classifier. Classifier overflow was pumped to a thickener which discharged by gravity to a drum filter. The filter cake was discarded along with the classifier sands collected in hoppers.

The mill used in this investigation was 30 in. ID by 47 in. long. The shell was made from ½ in. rolled plate and had eight half-round 1-in. radius lifters spaced evenly around the inside periphery. There were no liners in the mill. There were two riding rings on the exterior of the mill shell which sat in 1-ft diam flanged rollers, two on each side. The rollers rotated on ball bearings on stationary stub shafts. The mill and the rollers were mounted on top of a fabricated steel platform. The motor drive was installed underneath the platform, see Fig. 1.

Power for the mill was supplied by a 25-hp, 220-v dc motor controlled by a rheostat so that the speed could be varied. The motor was connected to a Jones speed reducer, which gave a 6.20 ratio. The gear reducer was connected to a stub shaft through a Baldwin-Lima-Hamilton SR4 strain gage torque meter pickup, capacity 1000 ft-lb. A 14-tooth sprocket on the stub shaft was connected by a chain to a 66-tooth sprocket around the outside of the mill. The stub shaft was supported rigidly by two ball-bearing pillow blocks bolted to the mill frame.

All the experiments were conducted with a rod charge which filled 40 pct of the mill volume. The rods were cold drawn steel round stock which had a hardness of 134 Brinell and analyzed 0.26 pct C, 0.008 pct P, and 0.037 pct S. Spectrochemical analysis showed the presence of 0.55 pct Mn, 0.17 pct Si, 0.03 pct Ni, 0.02 pct Cr, 0.003 pct Mo, and 0.08 pct Cu. Twelve rods were of 2½ in. diam, 22 of 2 in. diam, 32 of 1½ in., and 46 of 1 in. All were 45¼ in. long. Total weight of the rod charge was maintained as near 2850 lb as possible. At regular intervals during runs the charge was removed and weighed. Any loss was compensated for by the addition of rods.

The mill was operated with eight 3-in. diam openings, spaced evenly around the periphery of the discharge end. The total discharge area was 0.42 sq ft.

The power demand of the mill was determined from torque and speed measurements. The output of the torque pickup was amplified and the amplified signal permanently recorded by an Esterline-Angus recording milliammeter. Calibration of the torque meter was accomplished by application of static loads to the meter shaft so that a curve relating the foot-pounds of torque to the milliammeter reading could be plotted. Because the measurement of power by means of the torque meter was a direct measure of power supplied to the stub shaft that drives the mill, no corrections for motor or gear reducer efficiencies were necessary. The power demand was also determined by means of an Esterline-Angus recording wattmeter, connected to measure the input power to the motor. The demand was read directly from the recorder but had to be corrected for motor efficiency at the various speeds used during the investigation.

To obtain the mill speed, an ac tachometer generator was connected directly to the outboard shaft of the motor, and the voltage generated was transmitted through a rectifying system and recorded on a dc recording voltmeter. The recording voltmeter was calibrated with a Berkeley Events Per Unit Time Meter so that the meter was read directly in revolutions per minute.

Water from a constant head tank was added to the mill through Flowrators which were set to give the proper amount of water for the pulp density required.

At the beginning of each group of tests the operator checked the circuit for mechanical fitness and the instruments for proper adjustment. A feed rate was established by careful regulation of the Feedoweight; then the water flow was set by means of the Flowrator to give the calculated pulp density. Rotational speed of the mill was adjusted with the rheostat until the specified percentage of critical speed was indicated on the time meter. Feed rates were checked by timed samples.

Table I. Data for Rod Mill with End Peripheral Discharge 30 In. x 4 Ft.

Test No. (Feed)	Feed Rate, Lb per Hr (Waukesha Limestone)	Mill Speed, Rpm (Waukesha Limestone)	Pulp Density, Pct Solids (Waukesha Limestone)	P_{80} Microns (F_{80}=12,200)	K Intercept Microns (17,200)	M Slope (0.050)	Power Demand, Kw	Reduction Ratio	New Surface m^2 —x10^{-3} Hr	Work Index, Kw-Hr / Ton	New Surface Per Unit Energy m^2 —x10^{-3} Kw-Hr
1	1000	24	50	78	166	0.295	3.93	156.4	112.4	7.59	28.6
2	1000	29	50	73	153	0.302	4.97	167.1	112.3	9.27	22.6
3	1000	34	50	63	144	0.270	5.42	193.6	118.2	9.35	21.8
4	1000	39	50	70	161	0.268	6.31	174.3	114.1	11.58	18.1
5	1000	44	50	78	173	0.280	7.76	156.4	112.5	14.91	14.5
6	1000	24	60	71	149	0.301	4.42	171.8	112.6	8.15	25.5
7	1000	29	60	63	152	0.253	5.50	193.6	117.1	9.47	21.3
8	1000	34	60	59	139	0.260	5.99	206.8	119.1	10.03	19.9
9	1000	39	60	57	148	0.234	6.69	214.0	116.6	11.01	17.4
10	1000	44	60	69	172	0.244	6.89	176.8	112.9	12.49	16.4
11	1000	24	70	63	147	0.263	4.42	193.6	117.8	7.62	26.6
12	1000	29	70	58	141	0.251	5.27	210.3	118.7	8.73	22.5
13	1000	34	70	53	141	0.228	5.85	230.2	118.0	9.24	20.2
14	1000	39	70	55	146	0.227	6.49	221.8	119.0	10.29	18.3
15	1000	44	70	62	159	0.237	7.87	196.8	116.4	13.35	14.8
16	1000	24	80	79	187	0.259	3.96	154.4	110.7	7.70	27.9
17	1000	29	80	70	175	0.244	5.46	174.3	113.0	9.94	20.7
18	1000	34	80	71	173	0.250	5.88	171.8	112.8	10.85	19.2
19	1000	39	80	66	175	0.229	6.94	184.8	113.0	12.24	16.3
20	1000	44	80	97	235	0.252	5.68	125.8	105.0	12.29	18.5
21	2000	24	50	353	668	0.350	3.88	34.6	129.5	8.67	33.3
22	2000	29	50	207	379	0.369	4.67	58.9	151.6	7.66	32.5
23	2000	34	50	165	316	0.343	5.71	73.9	169.1	8.28	29.6
24	2000	39	50	152	300	0.328	6.40	80.3	177.2	8.84	27.7
25	2000	44	50	187	360	0.341	7.12	65.2	160.7	11.21	22.6
26	2000	24	60	261	496	0.348	4.21	46.7	145.7	7.85	34.6
27	2000	29	60	175	329	0.353	5.01	69.7	164.8	7.47	32.9
28	2000	34	60	148	293	0.327	5.54	82.4	179.6	7.51	32.4
29	2000	39	60	136	280	0.309	6.11	89.7	187.8	7.90	30.8
30	2000	44	60	188	400	0.296	6.51	64.9	173.2	10.16	26.6
31	2000	24	70	253	513	0.316	4.12	48.2	153.3	7.62	37.2
32	2000	29	70	168	341	0.315	4.94	72.6	177.1	7.11	35.9
33	2000	34	70	139	281	0.317	5.78	87.8	186.7	7.55	32.3
34	2000	39	70	139	292	0.301	6.44	87.8	188.6	8.38	29.3
35	2000	44	70	194	423	0.286	6.35	62.9	173.7	10.04	27.3
36	2000	24	80	302	612	0.316	3.94	40.4	145.9	8.05	37.0
37	2000	29	80	202	419	0.306	4.83	60.4	166.8	7.80	34.6
38	2000	34	80	166	364	0.284	5.77	73.5	182.4	8.33	31.6
39	2000	39	80	182	414	0.279	6.41	67.0	177.8	9.70	27.8
40	2000	44	80	207	465	0.276	6.05	58.9	172.5	9.88	28.5
41	4000	24	50	2480	4650	0.355	3.70	4.9	124.0	16.23	33.6
42	4000	29	50	1603	2967	0.362	4.46	7.6	139.9	14.01	31.4
43	4000	34	50	782	1450	0.361	5.41	15.6	185.6	10.03	34.4
44	4000	39	50	603	1123	0.359	6.00	20.2	200.5	9.70	33.4
45	4000	44	50	1260	2570	0.313	6.39	9.7	178.3	16.90	28.0
46	4000	24	60	2850	5380	0.351	3.64	3.5	115.0	19.70	31.6
47	4000	29	60	1225	2195	0.383	5.00	10.0	143.0	12.91	28.7
48	4000	34	60	637	1173	0.365	5.80	19.2	193.7	9.58	33.4
49	4000	39	60	570	1070	0.354	6.08	21.4	215.1	9.11	35.4
50	4000	44	60	1225	2600	0.296	6.13	10.0	202.7	14.86	33.1
51	4000	24	70	3120	5840	0.356	4.11	3.9	107.1	23.51	26.1
52	4000	29	70	1853	3495	0.352	4.07	6.6	137.7	14.25	33.8
53	4000	34	70	1065	1940	0.372	4.94	11.5	159.3	11.25	32.5
54	4000	39	70	780	1500	0.341	6.00	15.6	196.7	11.10	32.8
55	4000	44	70	2123	4350	0.311	7.09	5.7	152.8	28.07	21.5
56	4000	24	80	2620	5120	0.333	3.98	4.7	127.8	19.18	32.2
57	4000	29	80	1635	3110	0.347	4.37	7.5	146.0	13.90	33.4
58	4000	34	80	913	1757	0.341	5.51	13.4	179.7	11.78	32.7
59	4000	39	80	770	1487	0.339	6.47	15.8	192.3	12.20	29.8
60	4000	44	80	1620	3450	0.295	6.74	7.5	132.5	21.53	19.7
61	5000	24	50	4717	8587	0.372	3.91	2.6	106.1	28.15	27.2
62	5000	29	50	3160	5733	0.375	5.09	4.0	124.9	22.74	24.6
63	5000	34	50	1320	2393	0.375	5.59	9.2	179.9	12.02	32.2
64	5000	39	50	1013	1853	0.370	6.60	12.0	195.6	11.76	29.7
65	5000	44	50	2817	5283	0.355	6.16	4.3	143.2	24.93	23.3
66	5000	24	60	4105	7500	0.370	4.11	3.0	109.9	26.54	26.1
67	5000	29	60	2663	4837	0.374	4.78	4.6	134.6	18.40	28.2
68	5000	34	60	1547	2847	0.366	5.87	7.9	175.8	14.23	30.0
69	5000	39	60	1250	2397	0.343	6.40	9.8	205.2	13.26	32.0
70	5000	44	60	3335	6360	0.346	6.10	3.7	139.3	29.41	22.9
71	5000	24	70	4475	8600	0.342	4.20	2.7	121.8	29.52	28.9
72	5000	29	70	3065	6005	0.332	4.59	4.0	153.8	20.31	33.6
73	5000	34	70	1917	3683	0.342	5.43	6.4	177.3	15.67	32.7
74	5000	39	70	1373	2650	0.339	6.45	8.9	201.2	14.27	31.3
75	5000	44	70	3320	6667	0.320	6.30	3.7	156.6	30.24	24.9
76	5000	24	80	4450	8500	0.345	4.37	2.7	124.2	29.48	28.4
77	5000	29	80	3273	6217	0.348	5.19	3.7	138.3	24.57	26.7
78	5000	34	80	2270	4293	0.350	5.84	5.3	160.3	19.56	27.5
79	5000	39	80	1700	3353	0.329	6.40	7.2	192.1	16.78	30.0
80	5000	44	80	3100	6167	0.324	6.28	3.9	157.8	28.08	25.1

After the circuit was operating satisfactorily, the conditions were maintained for 45 min to insure equilibrium within the mill. During this time the pulp density was roughly checked by means of the pulp balance. Following the 45-min interval the tapes on the recorders were marked and were collected over a 10-min period. Samples of the mill discharge were also collected during this period. Three samples weighing about 30 lb each were taken at 3-min intervals. The entire flow of pulp from the mill was collected in tared tubs or buckets by removal of the launder between the mill and the classifier. Following this, a 40 to 50-lb timed sample of the entire flow of the feed was taken. The tests were continued by systematically changing the independent variables.

Table I contains the summary of the results of the 80 runs. The first three columns record the independent variables. The feed rate was obtained from the weights of the timed samples and the mill speeds were taken from the tape recording on the Events Per Unit Time Meter. Pulp densities were obtained

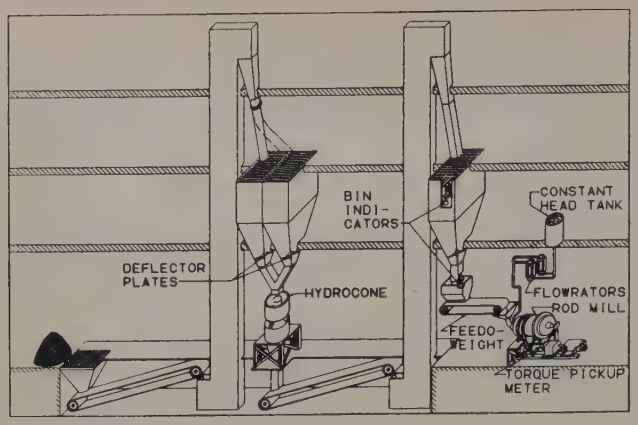


Fig. 2—Diagram of the crushing and rod milling circuit.

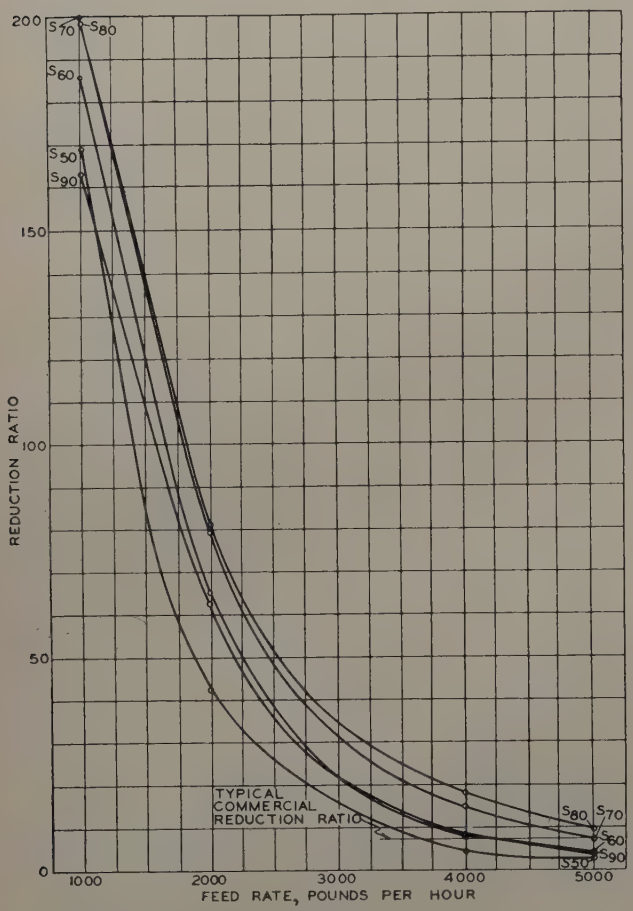


Fig. 3—Reduction ratio vs feed ratio for each mill speed. Averages for four pulp densities.

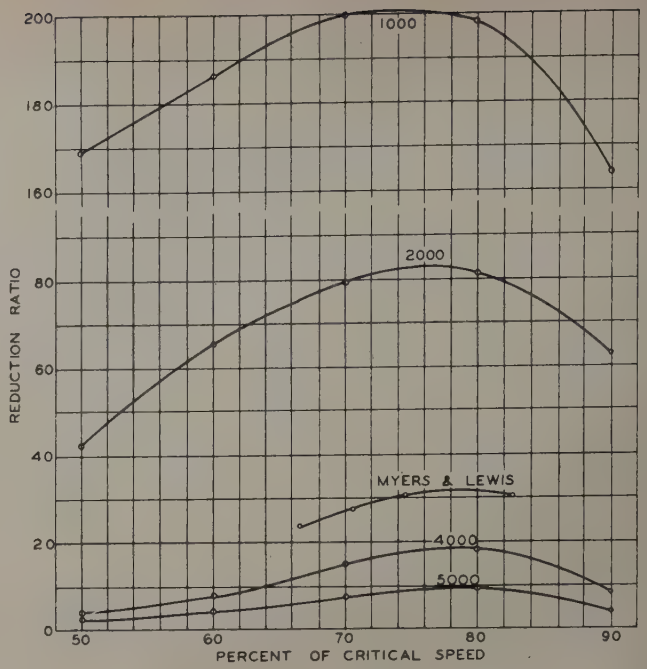


Fig. 4—Reduction ratio vs mill speed for each feed rate. Averages for four pulp densities.

from the samples of mill discharge that had been collected in tared buckets or tubs. The samples were weighed, dried, and weighed again to determine the moisture content. Average foot-pounds of torque was integrated on the torque meter tape by means of a planimeter. From these data, along with the speed, the power demands were calculated.

Dry sieve analyses of the feed samples and wet-and-dry analyses of the products were made. Results were plotted on log-log graphs with the log cumulative percent passing a given mesh as the ordinate and the log mesh size in microns as the abscissa according to the method described by Schuhmann.[5] Feed and product analyses were plotted on each graph. From each curve the slope, K intercept, and the F_{80} or the P_{80} were obtained.

From these data the reduction ratio and the new surface produced were calculated. The energy requirement per unit of new surface produced and the Bond work index[4] were also calculated, see Table I.

Surface areas were calculated from the sieve analyses according to the technique developed by Schuhmann[5] and modified by Bond.[6] Whether or not differences in the surface area of the feed and the product as calculated represent the absolute value of the new surface produced was considered incidental in this experiment, because only a measure-

Table II. Results of Variance Analyses. Three-Factor Analyses

Source of Variance	Components of Variance	Degrees of Freedom	Variance: Reduction Ratio	Variance: New Surface	Variance: Work Index	Variance: New Surface Per Unit Energy
F	$sd\,\sigma_f^2 + s\,\sigma_{fd}^2 + d\,\sigma_{fs}^2 + \sigma_o^2$	3	137,188.7	11,627.9	672.9	492.5
S	$fd\,\sigma_s^2 + f\,\sigma_{ds}^2 + d\,\sigma_{fs}^2 + \sigma_o^2$	4	1,506.2	6,378.8	153.4	128.4
D	$sf\,\sigma_d^2 + f\,\sigma_{ds}^2 + s\,\sigma_{fd}^2 + \sigma_o^2$	3	950.0	204.3	14.5	2.7
FxS	$d\,\sigma_{fs}^2 + \sigma_o^2$	12	250.3	953.6	48.4	23.6
FxD	$s\,\sigma_{fd}^2 + \sigma_o^2$	9	559.1	268.0	7.9	10.5
DxS	$f\,\sigma_{ds}^2 + \sigma_o^2$	12	24.3	85.8	3.3	6.8
Residual		36	25.1	70.0	2.4	4.0
Total		79				
Standard deviation			4.98	8.59	1.62	2.16
Relative deviation, pct			7.48	5.74	11.7	7.81

ment of the relative amount of work done during each test was desired for purposes of comparison.

Work indices were calculated from Bond's formula. This formula, as pointed out by R. A. Person of the Process Laboratory at Allis-Chalmers, can also be derived from the general equation reported by Walker, Lewis, McAdams, and Gilliland.[7] If n in their equation is replaced by 3/2, an equation identical to the Bond work index formula can be derived. Thus an energy size reduction relationship which is halfway between the Kick and Rittinger relationships is obtained.

Variance analyses were made of reduction ratio, work index, new surface, and new surface per unit energy to determine whether or not feed rate, mill speed, or pulp density or an interaction of feed rate, mill speed, or pulp density significantly affected them. Variance is defined as the sum of the squares of the deviations of a set of observations above and below their mean, divided by the number of observations. In the analysis of variance, averages of data at one level of an independent variable are compared with the averages of data obtained at other levels, and it is possible with this statistical technique to show whether or not the variations are significantly greater than chance variation or experimental error. The procedure used has been described by Brownlee[8] and by Dorenfeld.[9]

Variances obtained for the independent variables are shown on Table II. On this table, the letter *F* refers to feed rate, *S* to mill speed, and *D* to pulp density. The interactions of these variables are labeled *FxS*, *FxD*, and *DxS*. The residual variance is that portion of the total variability which cannot be attributed to the above factors or interactions, and is a measure of the experimental error. It may include a second order interaction, *FxDxS*. This interaction in a three-factor analysis is usually small. In testing the significance of the above variances, it has been assumed that the second order interaction does not exist or that it is not large. The components of variance, listed on Table II, aided in selecting tests of significance.

In calculating the variance of a finite sample, there is available only an estimate of the true mean of an infinite number of all possible results from which the sample is drawn. The sum of the squares of the deviations from the sample mean will be less than the sum of the squares of the deviations from the true mean. To correct for this and to obtain a better estimate of the variance, a divisor is used which is one less than the number of observations. This divisor is known as the degrees of freedom, see Table II.

The following general procedure was used for testing the significance of the variances shown on Table II. If a variance was less than the residual, it was considered nonexistent and was combined with the residual. *DxS*, *FxD*, and *FxS* were compared with the residual because these sources of variance included only the first order interaction and the residual. If the variance ratio reached 5 pct level of significance in tables of these ratios (95 pct certainty), the interaction was considered to have an effect; otherwise the variance attributed to the interaction was combined with the residual. At the 5 pct level the independent variable will have an effect on the dependent variable 95 times in 100.

The *DxS* interaction for each dependent variable was not significant, so it was combined with the residual. The *FxS* and *FxD* interactions, however,

Table III. Significant Variables. Three-Factor Analyses

Reduction Ratio	New Surface	Work Index	New Surface Per Unit Energy
*F**	*F**	*F**	*F**
*S***	*S***	*S*‡	*S*§
FxS†	*FxS*†	*FxS*†	*FxS*†
FxD†	*FxD***	*FxD***	*FxD*§§

* No test, but probably highly significant.
** Greater than 1 pct level.
† Greater than 0.1 pct level.
‡ Close to 5 pct level; may be significant.
§ 1 pct level.
§§ Slightly greater than 5 pct level.

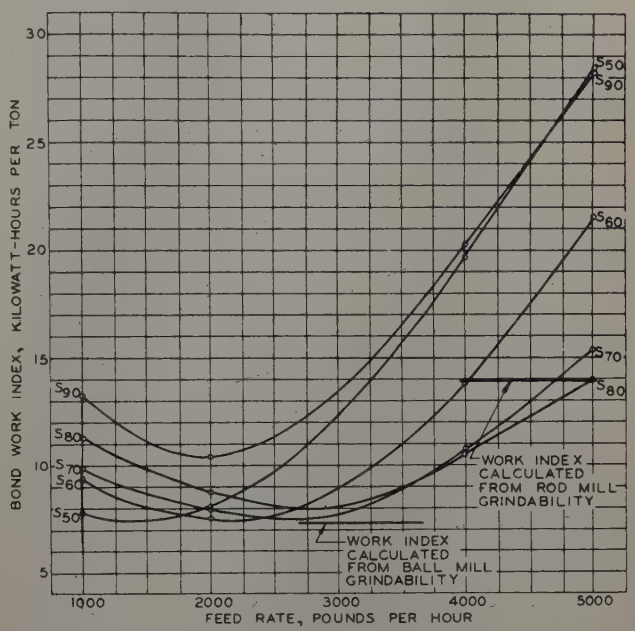


Fig. 5—Bond work index vs feed rate for each mill speed. Averages for four pulp densities.

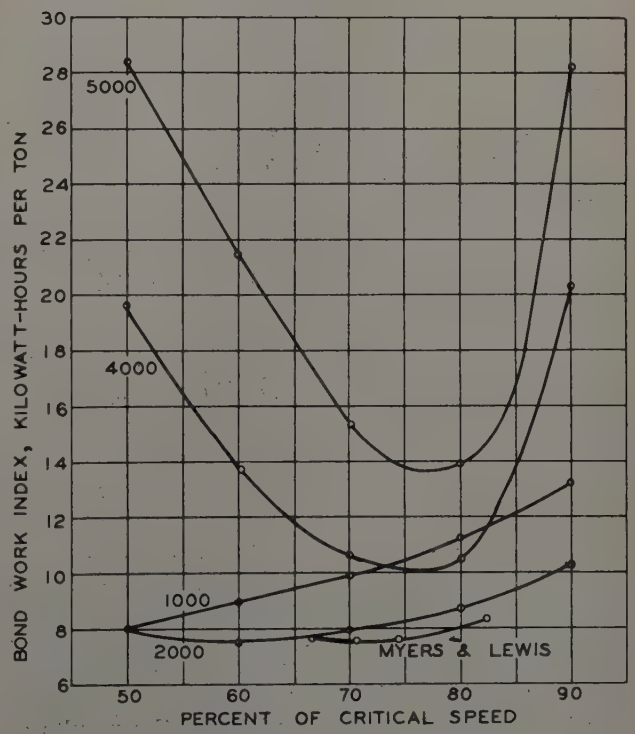


Fig. 6—Bond work index vs mill speed for each feed rate. Averages for four pulp densities.

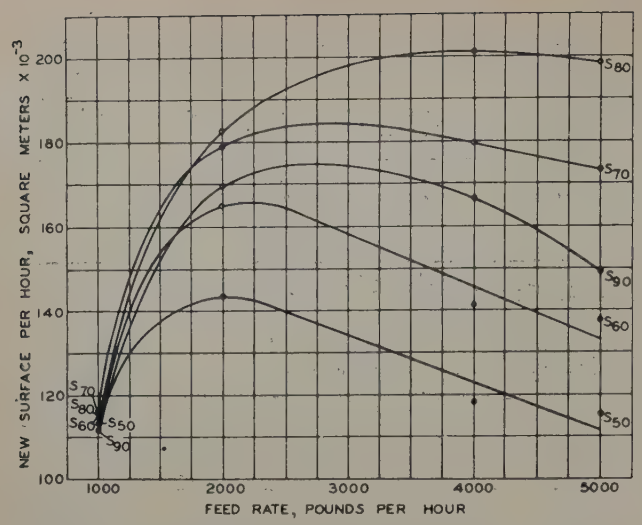


Fig. 7—New surface per hour vs feed rate for each mill speed. Averages for four pulp densities.

were. Consequently, S and D were compared with FxS and FxD respectively for significance.

It was not possible to make a test for the significance of F. However, because the variance attributed to F was large for all of the dependent variables, it appears that the effect of F must be significant in all cases. The significant variables and their levels of significance are shown on Table III.

The square root of the final combined residual was the standard deviation of any one experiment. These standard deviations, on Table II, show the magnitude of the error of the whole experiment. For comparisons of the error for one dependent variable with that of the other dependent variables, the relative deviation, or the deviation expressed as a percentage of the mean, is also shown.

Table IV. Results of Variance Analyses. Two-Factor Analyses

Source of Variance	Degrees of Freedom	Variance $F_{1/2}$	F_1	F_2	$F_{2\,1/2}$
Reduction Ratio					
S	4	1122.9	973.1	130.3	30.8
D	3	2434.9	171.9	17.2	3.2
Residual	12	77.4	18.9	2.3	1.0
Standard deviation		8.79	4.35	1.52	1.2
Relative deviation, pct		4.78	6.56	14.2	21.8
Work Index					
S	4	17.23	4.89	90.54	185.91
D	3	0.60	0.81	22.06	14.74
Residual	12	0.59	0.16	6.14	3.63
Standard deviation		0.772	0.399	2.48	1.91
Relative deviation, pct		7.48	4.7	16.5	8.9
New Surface					
S	4	18.3	956.4	4157.4	4107.6
D	3	44.9	290.7	537.3	135.3
Residual	12	3.1	11.2	207.8	73.6
Standard deviation		1.75	3.35	16.5	9.26
Relative deviation, pct		1.53	1.99	10.2	5.98
New Surface Per Unit Energy					
S	4	74.54	56.27	38.53	29.77
D	3	0.89	10.40	13.64	9.19
Residual	12	1.81	1.51	11.65	3.81
Standard deviation		1.28	1.229	3.47	2.21
Relative deviation, pct		6.22	3.94	11.2	7.81

Because it was not possible to test the significance of F, and because two interactions involving feed rate were significant, each analysis of variance was broken down into four two-factor analyses, one at each level of feed rate. In this manner it was possible to investigate the effect of mill speed and pulp density at each feed rate, and when the results at each level were compared with those at other levels, it was possible to determine how the effect of feed rate depended on mill speed and pulp density and, conversely, how the effect of mill speed and pulp density depended upon feed rate. Table IV shows variances obtained and Table V significant factors.

Table V. Significant Variables. Two-Factor Analyses

Item	Reduction Ratio	New Surface	Work Index	New Surface Per Unit Energy
$F_{1/2}$	S*	S†	S*	S*
	D*	D*		
F_1	S*	S*	S*	S*
	D**	D*	D‡	D†
F_2	S*	S*	S*	S§
	D†		D§	
$F_{2\,1/2}$	S*	S*	S*	S†
			D§§	

* Greater than 0.1 pct level.
** Between 1 and 0.1 pct levels.
† Greater than 1 pct level.
‡ Between 5 pct and 1 pct levels.
§ 5 pct level.
§§ Greater than 5 pct level.

Table V shows that pulp density was significant at several feed rates, whereas it was not significant in the three-factor analysis. Apparently this occurred because the magnitude of the variations resulting from changes in pulp density was small compared to the error for the entire experiment. With an analysis made at each level of feed rate, the variations attributed to pulp density became relatively more significant.

Significant Relationships

To determine what significant relationships were indicated by the variance analyses, the insignificant factor, density (three-factor analyses), was averaged out of the data, and curves were plotted of the dependent variables against feed rate for each level of mill speed, and against speed for each level of feed rate. To investigate the interactions of feed rate and mill speed, and feed rate and pulp density, the dependent variables were examined at each level of feed rate to determine how they varied with mill speed and pulp density when these independent variables were found to be significant (two-factor analyses). When an independent variable was not significant, it was averaged out of the data and the averages were examined for the relationship with the significant variable.

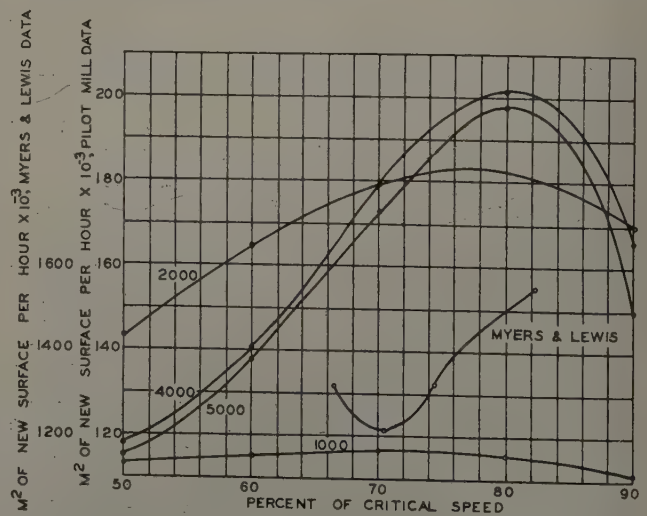

M² OF NEW SURFACE PER HOUR X 10⁻³ MYERS & LEWIS DATA
M² OF NEW SURFACE PER HOUR X 10⁻³ PILOT MILL DATA
PERCENT OF CRITICAL SPEED
MYERS & LEWIS
2000
4000
5000
1000

Fig. 8—New surface per hour vs mill speed for each feed rate. Averages for four pulp densities.

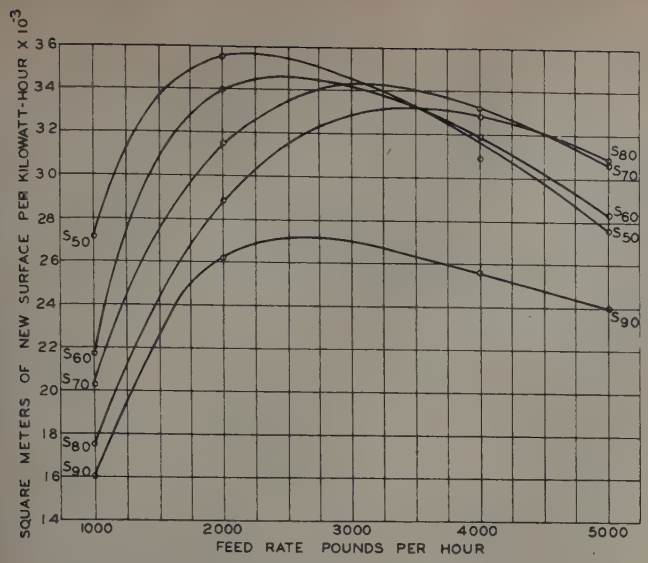


Fig. 9—New surface per kilowatt-hour vs feed rate for each mill speed. Averages for four pulp densities.

Reduction Ratio: Reduction ratio has been plotted against feed rate and speed on Figs. 3 and 4. Reduction ratio decreased rapidly as feed rate was increased, reaching maximum in the range of 70 to 80 pct of critical as the speed was increased. The *FxS* interaction can also be seen on these two figures. As feed was increased from 1000 to 5000 lb per hr, mill speed at which the maximum reduction ratio was obtained increased from 70 to 80 pct of critical.

The effect of the *FxD* interaction cannot be seen on these two curves because pulp density has been averaged out of the data. In Table V, density was found to be signfiicant at all levels of feed rate except at 5000 lb per hr, and the *FxD* interaction was shown to be significant on Table III. To comprehend this interaction, the original data on Table I were examined. It was found that at 1000 lb per hr the reduction ratio was maximum at a pulp density of 70 pct solids; at 2000 lb per hr, between 60 and 70 pct solids; and at 4000 lb per hr, at 60 pct solids. The effect of pulp density on reduction ratio at 5000 lb per hr was not significant, but at speeds of 70 and 80 pct of critical, definite maximums were obtained at 50 pct solids. Apparently as feed rate was increased from 1000 to 5000 lb per hr, the pulp density at which maximum reduction occurred was more dilute, from 70 pct solids at 1000 lb per hr to 50 pct solids at 5000 lb per hr.

A horizontal line corresponding to a typical commercial reduction has been drawn on Fig. 3. This reduction ratio, 7.3, corresponds to a reduction from 80 pct passing ½ in. to about 80 pct passing 10 mesh. At 50 pct of critical speed, this ratio could be obtained with a feed rate of about 3700 lb per hr; at 60 pct of critical speed, with a feed rate of about 4300 lb per hr; at 70 pct of critical speed, with a feed rate of 5000 lb per hr; and at 80 pct of critical, a feed rate greater than 5000 lb per hr would be necessary. In other words, for each 10 pct increase in the percentage of critical speed between 50 and 80, capacity increased roughly 15 pct.

Bond Work Index: Work index has been plotted against feed rate and speed on Figs. 5 and 6. The work index was at a minimum at feed rates from 1500 to 3000 lb per hr, depending upon the speed of the mill. It was at a minimum at speeds from 50 to 80 pct of critical, depending upon the feed rate. These curves show the *FxS* interaction in that as the feed rate was increased from 1000 to 5000 lb per hr, the speed required to obtain minimum work index increased from 50 to 80 pct of critical.

It was again necessary to resort to the original data to identify the *FxD* interaction. Pulp density was shown on Table V to have a significant effect at all feed rates except at 1000 lb per hr. At 2000 lb per hr minimum work index was obtained at 60 to 70 pct solids; at 4000 lb per hr, 50 to 60 pct solids; and at 5000 lb per hr, at 50 to 60 pct solids. Apparently as the feed rate is increased, the pulp density in the mill should be more dilute to obtain minimum work index.

The lowest work index for all operating conditions investigated was 7.5, which was obtained at 2000 lb per hr, at 60 pct of critical speed. The reduction ratio with these conditions was 65.4, which represents a reduction from 80 pct passing ½ in. to about 80 pct passing 65 mesh. This work index is practically the same as that calculated from the ball mill grindability at 100 mesh. A work index almost identical to that calculated from the rod mill grindability at 14 mesh, 13.9, was obtained at a feed rate of 5000 lb per hr and at 80 pct of critical speed. The reduction ratio with these conditions was 9.5, which is similar to the ratio obtained in many commercial installations.

For a reduction ratio of 7.3 (the ratio at which a 15 pct increase in capacity was noted with each 10 pct increase in the percentage of critical speed), the following work indices are indicated by the curves of Fig. 5. At 50 pct of critical speed and 3700 lb per hr, a work index of 17.0 is indicated. At 60 pct of critical and 4300 lb per hr, a work index of 15.8 is shown. At 70 pct of critical and 5000 lb per hr, a work index of 15.3 is shown. It appears that the increased capacity with increased speed was obtained at a greater efficiency.

New Surface: Figs. 7 and 8 show the effect of feed rate and mill speed on the production of new surface per hour. Maximum production of new surface per hour was obtained at feed rates from 2000 to 4000 lb per hr depending upon the mill speed. In the range corresponding to commercial rod milling

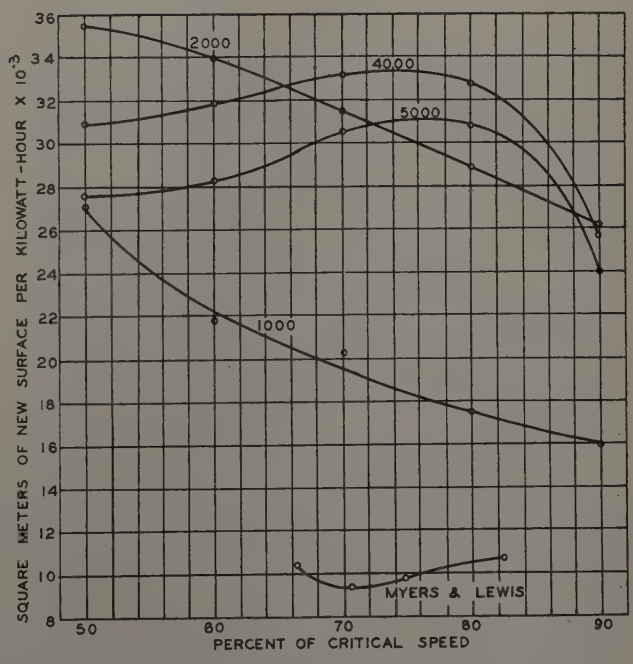


Fig. 10—New surface per kilowatt-hour vs mill speed for each feed rate. Averages for four pulp densities.

(feed rates of 3700 to 5000 lb per hr or more), it can be seen that the production of new surface decreased as the feed rate was increased. New surface was a maximum at speeds of 70 to 80 pct of critical depending upon the feed rate. Apparently the mill speed should be increased to 80 pct of critical as the feed rate is increased, for maximum production of new surface.

In Table V pulp density was found to be significant only at feed rates of 1000 and 2000 lb per hr. An examination of the experimental data for these significant effects revealed that at both 1000 and 2000 lb per hr, maximum production of new surface was obtained between 60 and 70 pct solids. Apparently the significant *FxD* interaction was only an indication that *D* was significant at 1000 and 2000 lb per hr, but not at 4000 and 5000 lb per hr. Even at low feed rates, the variation of new surface per hour with pulp density was small, so in general it can be said that the production of new surface was not greatly influenced by variations in the pulp density.

New Surface Per Unit Energy: New surface per unit energy has been plotted against feed rate and mill speed on Figs. 9 and 10. Maximum production of new surface per kilowatt-hour was obtained at feed rates from 2000 to 3500 lb per hr depending upon the mill speed. With this dependent variable, as with new surface, in the range of feed rates corresponding to commercial rod milling, the production of new surface per unit energy decreased as the feed rate increased. Maximum production of new surface per unit energy was obtained at 50 to 80 pct of critical speed depending upon the feed rate. The significance of the *FxS* interaction (Table III) is explained by the fact that as the feed rate was increased from 1000 to 5000 lb per hr, maximum production of new surface per kilowatt-hour was obtained at higher percentages of critical speed.

Pulp density was shown on Table V to be significant only at a feed rate of 2000 lb per hr. At this feed rate, maximum production per unit energy occurred at 60 to 70 pct solids, and the *FxD* interaction was significant only because the density effect reached significance at the 2000 lb per hr feed rate.

Variation of Work Index with Product Size: It was previously noted that the work index, calculated from Bond grindabilities for the Waukesha limestone used in this experiment, decreased as the product size decreased. In the factorial experiment, product size decreased with feed rate and the greatest variation for all dependent variables was attributed to feed rate. With most materials tested by the Bond grindability, the work index has either remained relatively constant as the product size decreased or it has increased slightly.[10] To determine whether or not the use of Waukesha limestone, with its characteristic of being easier to grind in the finer sizes, in any way invalidated the experiment or led to erroneous conclusions, the work index for this material determined by Bond grindabilities was plotted against product size. From this curve, the expected work index was obtained for the product size for each of the 80 runs of the experiment. The pilot mill data were then divided by the corresponding expected work index to remove the variation of work index with product size from the data.

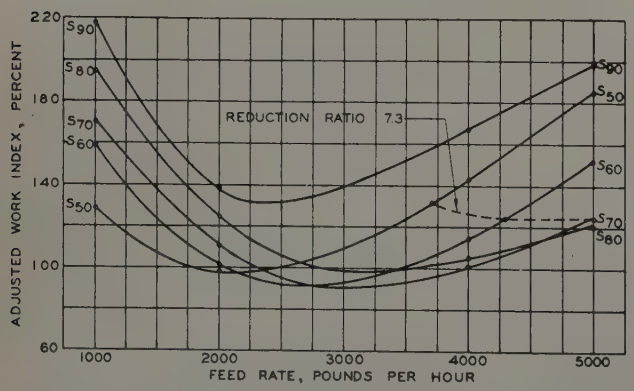


Fig. 11—Adjusted work index vs feed rate for each mill speed. Averages for four pulp densities.

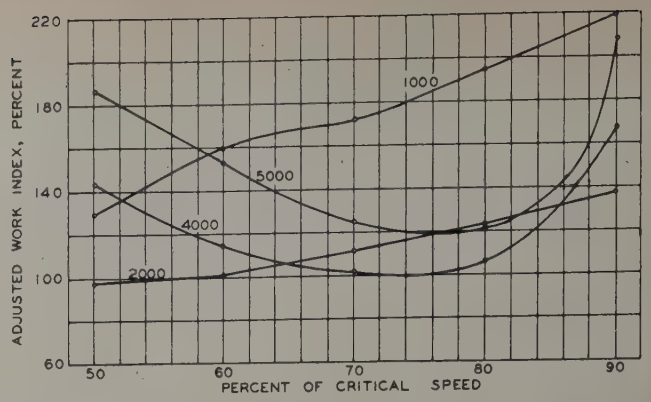


Fig. 12—Adjusted work index vs mill speed for each feed rate. Averages for four pulp densities.

A variance analysis was made of the normalized work indices. The same factors were found to be significant in this analysis as were found in the previous one. However, the relative deviation was more than double the previous value. This occurred probably because the removal of the variation with product size, formerly attributed to feed rate, decreased the total known variation and made the unknown variation or error relatively more important.

Because there was no test for *F* in the three-factor analysis, the analysis was broken down as before into four two-factor analyses, one at each level of feed rate. The only differences between these results and the results of the previous two-factor analyses for work index were: 1—there were greater relative deviations, and 2—less significance could be attributed to pulp density.

Density was averaged out of the normalized work index data and the averages were plotted against feed rate and mill speed. These curves are shown on Figs. 11 and 12.

Work index was a minimum at feed rates from 2000 to 3300 lb per hr depending upon the mill speed. As the feed rate increased, the speed at which the minimum occurred increased from 50 to 80 pct of critical. Minimum work index appeared to occur at more dilute pulps as the feed rate increased from 70 pct solids at 1000 lb per hr to 50 pct solids at 5000 lb per hr. However, the effect of pulp density only reached significance at 5000 lb per hr. Lowest work index for all operating conditions investigated was indicated at about 2700 lb per hr and at 60 pct of critical speed. This was a slightly higher feed rate but the same speed found from the unadjusted data.

An examination of Fig. 11 shows that for a reduction ratio of 7.3, the ratio at which a 15 pct increase in capacity was noted with each 10 pct increase in the percentage of critical speed, the work index appears to decrease slightly as the higher capacity at

this reduction ratio is realized. At least no decrease in efficiency is obtained as capacity is increased.

It appears that the use of Waukesha limestone with its characteristic of being easier to grind in the finer sizes has not invalidated the general conclusions drawn from this experimental work.

Reference to Published Data: A comparison was made between the results of this experiment and data presented by Myers and Lewis[11] who ground a copper ore from —1 in. to —10 mesh in open circuit in a 6x9-ft overflow rod mill at a pulp density of 79 pct solids. In the Myers and Lewis experiments, mill speed was varied from 66.5 to 82.4 pct of critical but all other conditions were held constant. Their data have been plotted against speed, see Figs. 4, 6, 8, and 10. Curves having a configuration similar to those obtained from this investigation were obtained for reduction ratio and work index, but the new surface and new surface per unit energy curves differed somewhat. Maximum reduction ratio occurred at about 80 pct of critical speed, and minimum work index occurred at 70 pct of critical speed. Minimum values were obtained at 70 pct of critical speed for new surface and new surface per unit energy, and these variables continued to increase at 80 pct of critical speed. Differences in some of the curves may perhaps be attributed to the difference in the type of mill discharge or to disparity in mill size.

Conclusions

The data presented in this paper were obtained from an experiment carried on in a relatively small rod mill grinding a limestone, and the relationships found may or may not be entirely representative of those prevailing in the large mills used in industry. Nevertheless, a comparison with published data for a 6x9-ft rod mill showed similar variations with speed, at least in regard to reduction ratio and work index.[11] It would seem reasonable that many of the principles involved in continuous open circuit grinding would be applicable irrespective of the size of the mill, and that only the magnitude of the relationships would differ.

In general, the effect of pulp density was not very great. Feed rate and mill speed were much more significant. However, the results showed that as the feed rate to the mill was increased, it was necessary to operate the mill with a more dilute pulp, 70 pct solids at 1000 lb per hr to 50 pct solids at 5000 lb per hr, to obtain the greatest reduction and the lowest work index. The effect of pulp density on the production of new surface and new surface per unit energy was seldom significant. At a few feed rates, however, maximum production of new surface was obtained between 60 and 70 pct solids.

It was found that as the feed rate to the mill increased, the mill should be operated at higher speeds up to 80 pct of critical to obtain maximum reduction, maximum production of new surface, minimum work index, and maximum production of new surface per unit energy.

The most efficient operating conditions for this rod mill occurred at a feed rate of 2000 lb per hr, a speed of 60 pct of critical, and a pulp density of 60 to 70 pct solids. With these conditions the work index was 7.5 and the reduction ratio was 65.4. When the data were adjusted to remove the variation in work index with product size, the most efficient conditions indicated by the adjusted data were a feed rate of 2700 lb per hr, and the same speed and pulp density indicated by the unadjusted data. With these conditions the work index was about 8.1 and the reduction ratio was about 30 (both figures obtained by interpolation). The product in either case was finer (80 pct passing 65 mesh or 80 pct passing 35 mesh) than that usually obtained from a commercial rod mill. These results indicate that on many ores the rod mill could probably be used to grind efficiently to finer product sizes than those ordinarily made in commercial operations. These results also suggest that further experimental work should be done to investigate the effect of increased feed size to the mill at these same operating conditions until a product as coarse as —8 or 10 mesh would be obtained.

In the range of feed rates at which a product size corresponding to a commercial rod mill reduction ratio of 7 to 10 was obtained, it was found that for each 10 pct increase in the percentage of critical speed between 50 and 80 pct, a corresponding increase in capacity of roughly 15 pct was realized at slightly increased efficiency. Best operating speed in this range of commercial reduction ratio was 80 pct of critical, best pulp density was 50 pct solids, and the work index was about 14.0 at a feed rate of 5000 lb per hr. This work index was practically the same as that calculated from a Bond rod mill grindability on the limestone at 14 mesh.

Whether the rod mill should be operated at the conditions which would give the lowest work index (7.5 to 8.1), or whether it should be operated at conditions which would give reduction ratios similar to those obtained in many commercial operations (7 to 10), is probably a matter of the economics of production costs compared with capital investment.

Acknowledgments

The authors acknowledge with thanks the assistance given by H. Wurcer, F. F. Miskell, E. O. Schneider, and L. E. Aguirre of the Process Section and by members of the Analytical, Metallurgical, Mechanical, and Physics Sections of the Research Laboratories. They also express thanks to J. B. Calkins, formerly with the Process Section and now with The Zonolite Co., and H. K. Ihrig, Vice President in Charge of Research, Allis-Chalmers Mfg. Co., for assistance in the preparation of the manuscript.

References

[1] S. D. Michaelson: Determination of Ball Mill Size from Grindability Data. *Trans.* AIME (1945) TP 1844.

[2] F. C. Bond and W. L. Maxson: Grindability and Grinding Characteristics of Ores. *Trans.* AIME (1939) **134**, p. 296.

[3] F. C. Bond, S. D. Michaelson, J. F. Myers: Rod-Milling Plant and Laboratory Data. *Trans.* AIME (1947) TP 2175, 11 pp.

[4] F. C. Bond: Third Theory of Comminution. *Trans.* AIME (1952) **193**, pp. 484-494.

[5] R. Schuhmann, Jr.: Principles of Comminution, I—Size Distribution and Surface Calculations. *Trans.* AIME (1940) TP 1189, 11 pp.

[6] F. C. Bond: Measuring Surface Area in Grinding. *Trans.* AIME (1941) TP 1296, 7 pp.

[7] W. H. Walker, W. K. Lewis, W. H. McAdams, and E. R. Gilliland: *Principles of Chemical Engineering*, pp. 254-255. McGraw-Hill Book Co., New York, 1937.

[8] K. A. Brownlee: *Industrial Experimentation.* Chemical Publishing Co., New York, 1949.

[9] A. C. Dorenfeld: Five Variable Flotation Tests Using Factorial Design. *Trans.* AIME (1951) **190**, p. 1073.

[10] F. C. Bond: Work Indexes Tabulated. *Trans.* AIME (1953) **196**, pp. 315-316.

[11] J. F. Myers and F. M. Lewis: Effects of Rod Mill Speed at Tennessee Copper Company. *Trans.* AIME (1949) TP 2585B, 2 pp.

A reminder that natural abrasives find an important place in modern industry. Various deposits of commercial value await mining operations in Canada.

Natural Abrasives in Canada

by T. H. Janes

NATURAL abrasives of some type are found in all countries of the world. In order of their hardness the principal natural abrasives are diamond, corundum, emery, and garnet, which are termed *high grade,* and the various forms of silica, including pumice, pumicite, ground feldspar, china clay and, most important, sandstone.

The properties qualifying materials for use as abrasives are hardness, toughness, grain shape and size, character of fracture, and purity or uniformity. For manufacture of bonded grain abrasives such as grinding wheels, the stability of the abrasive and its bonding characteristics are also important. No single property is paramount for all uses. Extreme hardness and toughness are needed for some applications, as in diamonds for drill bits, while for other purposes the capacity of the abrasive to break down slowly under use and to develop fresh cutting edges is of greatest importance, as with garnet for sandpaper. In dentifrices, soaps, and metal polishes, of course, hardness and toughness are objectionable.

First among the natural abrasives, **industrial diamonds** are essentially of three types: 1—*bort,* which includes off-color, flawed, or broken fragments unsuitable for gems; 2—*carbonado,* or black diamond, a very hard and extremely tough aggregate of very small diamond crystals; and 3—*ballas,* a very hard, tough globular mass of diamond crystals radiating from a common center. Bort comes from all diamond-producing centers, carbonados only from Brazil, and ballas chiefly from Brazil, although a few of this last group come from South Africa. By far the largest producer of industrial diamonds is the Belgian Congo; the Gold Coast, Angola, the Union of South Africa, and Sierra Leone supply most of the remainder. There is no production in Canada, which imports $6 to $9 million worth of industrial diamonds annually.

Industrial diamonds find innumerable uses in modern industry. They are used for diamond drill bits for the mining industry; in diamond dies for wire drawing; in diamond-tipped tools for truing abrasive wheels and for turning and boring hard rubber, fibers, and plastics; and in diamond-toothed saws for sawing stone, glass, and metals. High-speed tool steels, cemented carbides, and other hard, dense alloys can be cut, sharpened, or shaped efficiently only with diamond-tipped tools and diamond grinding wheels.

Second only to the diamond in hardness is **corundum**, an impure form of the ruby and sapphire gems consisting of alumina and oxygen (Al_2O_3) with impurities such as silica and ferric oxide. Corundum generally crystallizes from magmas rich in alumina and deficient in silica, as in the nepheline syenites of eastern Ontario. Grain corundum is used in the manufacture of grinding wheels; very coarse grain is used in *snagging* wheels. Both types of wheels are employed in the metal trades, where the hardness of corundum, coupled with its characteristic fracturing into sharp cutting edges, makes it an ideal cutting tool. The finest corundum (flour grades) is used for fine grinding of glass and high-precision lenses.

From 1900 to 1921 Canada was the world's leading producer of corundum. Following this period the deposits located in northern Transvaal of the Union of South Africa supplied more and more of the world's requirements, and since 1940 South Africa has provided almost the entire output, which has ranged between 2500 to 7000 tons a year during the last decade. Minor amounts have also been produced in Mozambique, India, and Nyassaland.

Opportunities for Mining Corundum

Corundum deposits in southeastern Ontario are of three types, which may be described as follows:

1—Scattered, irregularly-shaped deposits of coarse-grained corundum which could be mined by means of small pits. About 10 groups of such deposits are known. Although the tonnage of individual deposits of this type is not great, it has been estimated that several years' ore supply is available for a small tonnage operation. Deposits average about 9 pct corundum.

2—Large irregular deposits of coarse-grained corundum which would require mining by adit with possibly a scavenger operation on the remains of former surface deposits. The Craigmont deposit of this type produced about 20,000 tons of corundum concentrate during operations between 1900 and 1913. Most of the readily available surface ore was removed by operators during that time. Reserves of ore above road level have been estimated to average 7 pct corundum, but none of the so-called reserves have been blocked out, or even indicated, by diamond drilling. From 1944 to 1946, 2025 tons of

T. H. JANES is Engineer, Industrial Minerals Div., Mines Branch, Ottawa, Ont.

Discussion on this paper, TP 3899H, may be sent (2 copies) to AIME before Dec. 31, 1954. Manuscript, Jan. 21, 1954. New York Meeting, February 1954.

Fig. 1—Locations of natural abrasives in Canada are indicated by numbers 1 through 12 as follows. 1—Corundum, Craigmont. 2—Garnet, River Valley. 3—Garnet, Labelle. 4—Quartzite, Manitoulin. 5—Sandstone, Joyceville. 6—Sandstone, St. Canut. 7—Grindstones, Sackville. 8—Grindstones, Clifton. 9—Grinding pebbles, Elkwater Lake. 10—Pumicite, Waldeck. 11—Pumicite, Deadman River. 12—Diatomite, Quesnel.

corundum were recovered from the tailings of former operations that averaged 2.96 pct corundum.

3—A large body of fine-grained corundum, averaging about 5 pct corundum, disseminated throughout a nepheline-feldspar-mica gneiss. A considerable tonnage of this material is available in the Monteagle deposit near Bancroft, and in 1951 tests for the separation and recovery of corundum, nepheline, and muscovite mica were undertaken by Ortona Gold Mines Ltd.

The southeastern Ontario corundum-bearing zones may be divided roughly into three belts: the main or northern belt, the Methuen-Burleigh or middle belt, and the Lanark-Frontenac or southern belt. The rocks comprising these belts are, in general, of four types, but in many cases one type merges into another. Corundum-bearing rocks in the northern belt are syenites, syenite-pegmatites, nepheline syenites, and anorthosites. It is believed that corundum is one of the last constituents to crystallize out from the molten magma and that the whole of the excess of the alumina separates out as corundum, provided that the iron, magnesia, and silica present are low. Corundum does not occur in the immediate vicinity of free silica.

Canadian imports of graded grain corundum, according to the Dominion Bureau of Statistics, amounted to 162 tons valued at $43,450 in 1953 compared to 125 tons valued at $31,066 in 1952.

In northern Transvaal individual deposits vary widely in size but are invariably associated with pegmatite intrusions into basic rocks. Until recent years most of the production has been obtained from eluvial deposits resulting from weathering of pegmatites where the corundum is recovered by hand-cobbing or by gravity separation methods. Recovery from reef deposits is usually confined to those in which the rock has been weathered. After crushing the corundum is recovered as outlined above. Boulder corundum, carrying 60 to 70 pct corundum, undergoes no treatment before shipment other than breaking into sizes convenient for bulk handling. Many deposits of corundum occur in Southern Rhodesia, Nyassaland, and Mozambique. The price of corundum concentrate varies between $85 to $110 per ton according to grade. A minimum corundum (Al_2O_3) content of 90 pct is desired.

Emery is an intimate mixture of corundum and magnetite, with or without hematite, varying in hardness and toughness according to the amount of iron oxides present. Emery is massive, nearly opaque, and dark gray to blue-black with a reddish tinge depending on the amount of hematite present. It usually occurs in crystalline limestones and

schists, often with chlorite in hornblende schists in association with peridotite and diorite. The iron oxide is physically inseparable from the corundum, and while it detracts from the efficiency of emery as an abrasive, it adds to its polishing action. Grain shape of the emery is more or less round and for this reason the cutting action of the emery is slight.

Grades of Emery, Sources of Supply, and Imports of Canada

The three main emery-producing countries of the world are Greece, Turkey, and the United States. Grecian (Naxos) emery contains about 65 pct corundum and about 25 pct magnetite. In corundum content and quality Turkish emery approaches that produced in Greece. American emery, most of which comes from the state of New York, is the softest of the three and contains about 45 pct iron oxides.

Canadian imports of emery powder, grains, and grits for abrasive purposes were valued at $95,110 in 1953 compared to $54,566 in 1952. A large part of the U. S. production of about 5000 tons a year is used in concrete and asphalt floors in industrial plants because of its marked resistance to wear and its non-skid nature. The balance of the output, together with imports from Greece and Turkey, is used in abrasive products such as coated papers, abrasive sticks, and grinding wheels. The price of American first-grade emery, f.o.b., New York, is about $12 per ton. Grain emery, f.o.b., Pennsylvania, is quoted at 10¢ per lb for Turkish and Naxos grain, 6½¢ per lb for American grain.

Garnet is the name given a certain group of minerals possessing similar physical properties and crystal forms although their chemical compositions vary widely. The garnet group comprises seven different species, all of which are silicates of aluminum, calcium, iron, magnesium, manganese, or chromium, the different metals being replaceable one by the other. The garnets may vary widely in color, but almandite (iron aluminum silicate), the commonest, and andradite (calcium iron silicate) range in color from deep red through brown to black.

Garnets occur as accessory minerals in a large variety of rocks all over the world but are particularly common in gneisses and schists. When they are present in sufficient quantities the rocks are designated as garnet gneisses or schists. They also occur as contact metamorphic deposits in crystalline limestones, pegmatites, serpentines, and other rocks. Hornblende and mica are the usual associated minerals, quartz, feldspar, and pyroxene being present in lesser amounts. Garnets are *high-temperature* minerals and are usually found around intrusive contacts or vein deposits of high-temperature origin. Because of their resistance to weathering and erosion, garnets are often found as a detritus of crystals in the immediate vicinity of the original rocks, or as rounded grains in river or sea sands.

Garnet in the World Market

Although garnet is a common constituent of rocks, large workable deposits suitable for manufacture of garnet paper and cloth are scarce. Commercial production of this type of garnet has been confined chiefly to the United States, and even here successful operations on a large scale have been limited to one area in New York State. New York almandite garnet has incipient lamellar parting planes which cause it to break into thin, sharp-edged plates. This unique physical property renders it the world-standard garnet for manufacture of garnet paper and cloth. Other garnets may possess equal toughness and be just as suitable for less important abrasive uses, but generally they are not acceptable substitutes for New York garnet. World production and consumption of garnet for this use ranges from 7000 to 9000 tons a year, and this together with competition from artificial abrasives has severely restricted the search for garnet and the development of known deposits. In recent years garnet has been recovered from garnet sands in Idaho for use in the sandblasting and metal spraying industries. About 4000 tons were reported sold in the United States for these uses in 1951.

In New York State, Barton Mines Corp. mines a deposit of garnet on Gore Mountain, 5 miles west of the village of North Creek. The orebody is described as being about ¾ miles long and varying in width from 50 to 300 ft. The almandite garnet occurs in a metamorphic rock in which hornblende is the principal gangue mineral, associated with feldspars and hypersthene and smaller amounts of apatite, biotite, and pyrite. The garnet occurs mostly as imperfectly developed crystals varying from a fraction of an inch to more than a foot in diameter and constitutes from 5 to 20 pct of the orebody, which averages about 10 to 12 pct garnet. The ore is removed by quarry benching methods and the garnet recovered by a sink-float separation, ferro-silicon being used as the heavy medium.

Canadian production of garnet has been negligible. There are many occurrences in gneisses and schists in all parts of Canada, but the garnets are generally of small size and badly fractured. Niagara Garnet Co. Ltd., Sturgeon Falls, Ont., intermittently operates a deposit near River Valley in which crystals range in size from marbles to those of 4-in. diam. Open-cut methods are used for mining. After preliminary crushing the material is passed through trommel screens, and the oversize, carrying about 60 pct garnet, is trucked to the mill at Sturgeon Falls for further crushing, concentration, and sizing. Minor amounts of garnet grain and flour grades of garnet have been marketed during recent years. The garnets occur in gneiss associated with hornblende, and flake mica constitutes the main accessory mineral. Several years ago a high-grade garnet occurrence in massive pyrrhotite was mined near Labelle, Que. The deposit was of minor extent and was quickly mined out.

Probably about 90 pct of the garnet produced is employed for the manufacture of abrasive-coated papers and cloths. The remainder is used in the form of loose grains for surfacing and polishing relatively soft stones, for some metal-spraying and sandblasting operations, and in minor amounts for surfacing plate glass.

Garnet production in the United States in 1951, as reported by the U. S. Bureau of Mines, reached an all time high of 14,050 short tons, valued at $1,246,947. The three Canadian manufacturers of coated paper and cloth use about 450 tons of graded garnet grain annually.

The current price of ungraded garnet suitable for sandpapers is about $93 per ton f.o.b. New York. Prices of graded garnet grain range up to $160 per ton. Superfine powders in the 5 to 10 micron size for lens grinding sell at about $200 per ton.

Silica in many forms is used for abrasive purposes, including quartz and quartzite, sandstone, flint, amorphous silica, tripoli, diatomite, and rotten-

stone. Its particular abrasive use depends mainly on sharpness of the grains.

Crushed and graded **quartz** is used for abrasive backing of the cheapest of all abrasive-coated papers, *flint* sandpapers, which are used extensively for working with soft woods. Garnet and quartz papers may be used for the same type of work but generally the quartz, or flint-papers, are used for soft woods and the garnet papers for hard woods. True flint papers are used extensively in Europe for both types of work, as true flint has better cutting qualities and longer life than ordinary quartz. Powdered quartz and silt are sometimes used for scouring compounds and for harsh metal polishes.

Silica sand from sandstone and beach sands is used extensively for sand blasting, metal spraying, initial grinding or surfacing of plate glass, and cutting with gang saws on stone. Although grains are usually spherical, they may be sharp if they were not moved far during the stage of deposition in the original sediments. Natural silica sands are formed by the breakdown of sandstone, quartz, or quartzite and occur as beaches along lakes and oceans. They consist essentially of quartz grains with varying quantities of impurities. There is no production of abrasive sand in Canada and requirements are obtained from the United States.

In sand-blasting or metal-spraying operations the sand must be absolutely dry and free from dust. It can be used over and over again until the grains become too fine. The following size ranges for sand blasting have been outlined: sand No. 1, 20 to 35 mesh; No. 2, 10 to 28 mesh; No. 3, 6 to 10 mesh; No. 4, 4 to 8 mesh. No. 1 is used for light work where comparatively smooth finish is desired: for finishing brass, making automobile castings, frosting glass, and removing paint. No. 2 is used for similar work of a heavier nature. Sands No. 3 and 4 are used for cleaning heavy cast iron and steel work.

Apparently no Canadian sand or sandstone has been used for sand blasting or for coating abrasive papers. In some trials it was found that after processing the material was too fine-grained or fractured too easily in use. The Lorrain quartzite which is quarried by Canadian Silica Corp. Ltd. on Manitoulin Island for the manufacture of artificial abrasives could be a source of sand for this purpose.

Canadian Consumption of Silica Sand

About 100,000 tons of silica sand valued roughly at $600,000 are consumed annually in Canada in the manufacture of artificial abrasives. For the manufacture of silicon carbide, specifications vary widely from company to company, but in general the material would be between 35 and 60 mesh and contain a minimum of 98.5 pct SiO_2 with a maxima of 0.5 pct aluminum oxide, 0.035 pct ferric oxide, and 0.2 calcium and magnesium oxides combined. Grain shape is not important in silica to be used for the manufacture of silicon carbide.

Silica sand from a sandstone deposit at St. Canut, in Two Mountains County, Que., is quarried by the Canadian Carborundum Co. Ltd. for making silicon carbide in the Shawinigan Falls plant. A Potsdam sandstone is quarried near Joyceville north of Kingston, Ont., by Kingston Silica Mines Ltd. for use in steel foundries and for manufacturing artificial abrasives. Lorrain quartzite, quarried by two companies, one on Manitoulin Island and one nearby on the mainland, is used in the manufacture of silicon and ferro-silicon. These are the current Canadian sources of silica. Most of the silica sand of high quality is imported and, including sand for glass manufacturers, amounts to approximately 700,000 tons annually.

Grindstone production in Canada in recent years has been negligible. Material suitable for grindstones, oilstones, millstones, chaser stones, and other types of sharpening stones occurs in sandstone beds of Nova Scotia, New Brunswick, and the coast of British Columbia. Many years ago output from quarries operated in these provinces was considerable, but competition from artificial abrasives has all but eliminated current demand.

Sandstone suitable for grinding or sharpening purposes must possess uniform hardness and a sharp and even grain and must be free from clay or other impurities. The bond or cement which holds the sand particles together has an important bearing on the quality of the stone. Cements usually consist of limonite, calcite, clay, and quartz and may occur individually as the sole bond or be mixed in varying proportions. Stones having too much clay bond absorb water readily and crumble away. The size of the grit determines the ultimate use of a sandstone for grinding purposes, since the coarser the grit the faster and rougher the cutting.

The only Canadian producer of grindstones, Read Stone Co. Ltd. of Sackville, N. B., ships small quantities of grindstones each year. Production valued at $10,000 was reported in 1950. The company obtains its stone from quarries near Stonehaven. The Bay of Chaleur Grindstone Co. of Clifton, N. B., reported small production of stone up to 1950 but in recent years has not operated its quarry situated on the Bay of Chaleur near Grand Anse.

Statistics on the imports of natural abrasive stones into Canada from the United States are difficult to obtain, as they are lumped with data for stones made of artificial abrasives. U. S. export figures show that Canada, in 1952, imported grindstones valued at $27,376 and whetstones, scythestones, sticks, and other small sharpening stones valued at $21,798.

Pulpstones of natural sandstone used in the grinders of pulp mills have been almost displaced by artificial abrasive in the form of built-up segmental pulpstones of bonded silicon carbide grit. Pulpstones were made from sandstones having a similar texture to those used for grindstones and the same quarry might furnish both types of stones. However, a sandstone bed from which pulpstones can be made must have a thickness of at least 3 ft if even the smallest pulpstones are to be obtained.

The life of a natural pulpstone might be from 3 to 20 months, whereas that of the segmental silicon carbide stones might be from 4 to 7 years. Natural pulpstones have not been produced in Canada for many years, but they have been produced in Northumberland and Westmoreland Counties in New Brunswick and in Gabriola and Newcastle Islands off the east coast of Vancouver Island, B. C. About 800 artificial pulpstones are in use in Canadian pulp and paper mills and about 300 are in stock at the various mills. Most of these have been made by Norton Co. of Canada Ltd., Hamilton, Ont., which also supplies some for export. Artificial pulpstones supplied by Canadian Carborundum Co. Ltd. to the pulp and paper industry in Canada are made in its United States plant and imported into Canada.

Flint is an extremely hard chalcedonic variety of silica (SiO_2), light to dark gray in color, with a prominent conchoidal fracture. Flint pebbles have

long been used for grinding ceramic materials and nonmetallic minerals where iron contamination from the usual steel grinding balls or liners would prove detrimental. Danish flint pebbles ranging from 1 to nearly 8 in. are found on the shores of Greenland and marketed throughout Europe and America. Domestic natural flint pebbles, quartz, and quartzite are often substituted for *true flint* pebbles.

Grinding pebbles in minor quantities are produced in Alberta, where W. May ships a few carloads each year from deposits near Elkwater Lake to several Canadian mining companies. Sometimes tube mill liners, when very low iron content of the product is necessary, are also made of blocks or bricks of either natural or artificially prepared materials free of iron.

Certain silicates are used for abrasive purposes, particularly glassy volcanic rocks such as pumice and volcanic dust, or pumicite. **Pumice,** a silicate of aluminum, occurs as lump or gravel in the vicinity of volcanoes and is usually white or light gray in color. Its cutting or abrasive quality is due mainly to thin partitions of glass between the walls of the cells. Lump pumice varies in composition and contains impurities, such as feldspar and hornblende, which lessen its value as an abrasive because they scratch articles being polished. A good pumice contains 65 to 75 pct silica, 12 to 15 pct alumina, less than 3 pct combined alkalies (sodium and potassium oxides), under 3 pct calcium and magnesium oxide, less than 3 pct iron oxide, and small amounts of other oxides.

Pumicite, sometimes called volcanic dust or ground pumice, is similar to pumice in composition and has similar applications. Pumicite is a natural glass or silicate thrown into the air during volcanic eruption and carried in the air to settle ultimately in beds that may be hundreds of miles from its original source. Of white to grayish color, it occurs as a finely divided powder composed of small, sharp, angular fragments of volcanic glass. Many of these fragments are striated, which helps to distinguish pumicite under the microscope from other forms of silica or silicates.

The most important use for pumicite is in scouring compounds and cleansers. It is made into hand soaps, is sometimes employed as a carrier for insecticides, and is used increasingly as an extender in concrete. Pumice aggregate is imported into British Columbia from nearby Oregon and Washington for making lightweight building blocks at several plants in and near Vancouver.

Volcanic dust or pumicite occurs in many localities in central and western Canada. Extensive beds of volcanic dust occur near Waldeck about 11 miles northeast of Swift Current in Saskatchewan, the deposit consisting of loosely compacted, finely divided material which is generally light buff in color but sometimes nearly white. A pure white pumicite occurs in the Deadman River region about 25 miles north of Savona in British Columbia. Between tuff beds varying in thickness from 10 to 30 ft, horizontal beds of pure white ash 8 to 12 ft thick occur. Here again, transportation difficulties have hindered the development of these deposits. Several other occurrences in Saskatchewan and British Columbia are on record.

Extensive pumicite beds occur in Nebraska and Kansas in the United States. The majority of the beds are wind-blown deposits 6 to 10 ft thick, covered with overburden. Imports of pumice and pumicite into Canada, mainly from the United States, grouped together with lava and calcareous tufa, were valued at $110,369 in 1952 compared with $128,957 in 1951. The pumice aggregate imported into British Columbia from Oregon and Washington is laid down at Vancouver from $6 to $9 a short ton. Prices for pumice and pumicite vary widely and are dependent upon availability of supply, the use to which the material is put, and the quantities required. Recent quotations on pumicite were from 3¢ to 5¢ per lb, and for good quality pumice 6¢ to 8¢ per lb, f.o.b. New York or Chicago.

Diatomite consists of microscopic siliceous skeleton remains of diatoms, a form of algae. It has the appearance of chalk when dry but is extremely light in weight. When pure it contains up to 96 pct silica in the form of the diatom skeletons.

Diatomite is used for filtration, coating ammonium nitrate prills, insulating against heat, cold, and sound, and admixing concrete. It has a minor use as an abrasive in paste and liquid metal polishes, and in dental powders and pastes, where grit, with consequent scratching, can not be tolerated.

There are more than 300 known occurrences of diatomite in Canada, but these are mainly of the bog type and the processed material would not be suitable for the major uses. There are large dry compact beds of diatomite near Quesnel, B. C., but transportation difficulties have hindered their development.

Known to the trade as soft silicas, tripoli, microcrystalline silica, and rottenstone are fine-grained and porous. There is no production of these materials in Canada and they are not listed separately in import figures but are included in miscellaneous imports.

Tripoli is a form of silica closely resembling diatomite but of entirely different origin. It was first quarried in Missouri and because of its close resemblance to material occurring at Tripoli, North Africa, it was called *tripoli* although the African product is true diatomite. Under high magnification the grains in tripoli have a mostly globular or spongy appearance in contrast to the striated, sharp, glass-like fragments in volcanic dust or the absence of all diatom structure in diatomite. It is used in the form of grease bricks or compositions for buffing and polishing metals and plated products. It is also used in the manufacture of some scouring and cleaning powders and soaps.

Microcrystalline silica appears to be a decomposition of chert (a form of flint) and generally occurs mixed with unaltered chert. It is usually pure white but may be stained by iron oxides to cream yellow or rose. Microcrystalline silica comes mainly from southwestern Illinois, Missouri, and Tennessee and like tripoli is used in buffing and polishing compounds. Both tripoli and microcrystalline silica are used in paint as fillers, in insecticides as carriers, and in the manufacture of rubber.

Rottenstone is a residual product derived from weathering and decaying of a siliceous argillaceous limestone where the calcium carbonate and other impurities have been leached out to leave a siliceous skeleton. It occurs as a soft, friable, fine-textured earthy mass usually greyish in color. Produced at Antes Forte, Lycoming County, Pa., it is used as a base for automobile and furniture polishes.

The total production value of these three substances in the United States amounts to $200,000 annually (about 12,000 tons).

Ground feldspar, chalk, china clay, and bath brick are used in minor amounts as ingredients in scouring and cleansing compounds and in fine polishing powders. They are also mild abrasives for hand polishing plated ware. Chalk (calcium carbonate) is a soft, compact, fine-grained white limestone composed of the calcareous remains of small marine shells. Bath brick is made from a very fine-grained quartzose clay found in England.

Summary

The list of natural abrasives might include any mineral capable of abrasive action. Natural abrasives occur throughout the world, and frequently local deposits of abrasive material render it convenient to use that material for a purpose to which some other type of abrasive is more suited. Economics often determine which abrasive is to be used for a particular job, but there are some applications to which only a particular abrasive may be applied.

References

V. L. Eardley-Wilmot: Abrasives, Pt. I. Siliceous Abrasives. Mines Branch, Dept. of Mines, Ottawa, Pub. No. 673 (1927).

V. L. Eardley-Wilmot: Abrasives, Pt. II, Corundum, Emery, and Diamond. Mines Branch, Dept. of Mines, Ottawa, Pub. No. 675 (1927).

V. L. Eardley-Wilmot: Abrasives, Pt. III, Garnet. Mines Branch, Dept. of Mines, Ottawa, Pub. No. 677 (1927).

V. L. Eardley-Wilmot: Artificial Abrasives and Products. Mines Branch, Dept. of Mines, Ottawa, Pub. No. 699 (1930).

Minerals Yearbook, U.S. Bureau of Mines 1948, to date.

R. B. Ladoo: *Non-Metallic Minerals: Occurrence, Preparation, and Utilization.* McGraw-Hill Book Co., New York.

Industrial Minerals and Rocks: Seeley W. Mudd Series, AIME, New York, 1949.

Mines Branch Publications of the Department of Mines and Technical Surveys, Ottawa.

John S. Crandall: Production and Marketing of Garnet Abrasive Sands. *Trans.* AIME (May 1950) **187.**

Evaluating the Performance of a Cleaning Unit

by J. Visman

A simplified method of assessing the characteristics of a cleaning unit, including washability curves, yield figures, ash error, separating gravities, and error curve.

FOR more than 25 years evaluations of coal cleaning units have appeared in many publications, and during this time considerable progress has been made in developing the theory and techniques of calculating washery results. A variety of characteristics have been defined over the years, many receiving general recognition during the first post-war Coal Preparation Conference in Paris in 1950. Coal preparation has become a science, and the approach to preparation problems is being revolutionized. Formerly, for example, if a coal operator wanted to know the performance of a given cleaning unit for a particular grade of his run-of-mine, he either shipped a carload to the manufacturer for a test run or relied on a report of a test carried out on a coal similar to the one he intended to clean. In modern practice the expensive, time-consuming test work generally can be avoided if the washability curves of the coal in question and the error curve of the cleaner are known. Normally a guarantee can be given on the strength of this information. General acceptance of modern standards has thus reduced the need for pilot-scale models of existing types of coal cleaners. Needless to say, the method requires exhaustive and accurate information on the cleaning unit and on all the additional circumstances affecting its results, information that ultimately can be secured only from actual tests made in the laboratory or in the washery.

It is not the purpose of the present discussion to compare the merits of various characteristics, but to describe the general method of calculation, including a method of obtaining data with a minimum of laboratory work.

All data presented here were derived from tests made at the plant with portable field equipment, using small quantities of varsol, carbon tetrachloride, and bromoform. It will be explained later that the use of standard fractional ash contents designated *normalized* ash contents aids considerably in reducing the routine testing of coal cleaners. Like any other statistical procedure, this method has limitations, and it is not recommended for any work in connection with guarantees. Even a varying commodity such as coal, however, will follow the law of large numbers, which is the basic law of statistics, thus allowing simplification of test work without appreciable loss of accuracy.

The characteristics needed for evaluating cleaner performance are normally derived from a float-sink analysis of samples of the clean coal and the refuse, followed by ash determinations of the various fractions. Usually a sample of feed is also collected to find its ash content and to make a single specific gravity cut, both in connection with determination

J. VISMAN is Preparation Engineer, Office of the Fuels Div., Calgary, Alberta, Dept. of Mines and Technical Surveys, Ottawa, Ont., Canada.

Discussion on this paper, TP 3898F, may be sent (2 copies) to AIME before Dec. 30, 1954. Manuscript, March 8, 1954. Joint Session CIMM and Coal Div., AIME, February 1954.

of yield. This test work is time-consuming, especially for larger sizes of coal, and therefore is seldom done at the plant. Even the manufacturers of coal-cleaning equipment sometimes dispense with a float-sink test, basing their guarantees on an ash reduction figure and the yield. The last-named figures are generally considered insufficient information, as they change according to coal composition and density of separation. No single characteristic, in fact, adequately describes the efficiency of a coal cleaner. The one that comes closest is the *error curve,* with its derivatives, the *separating gravity,* the *probable error,* and the *imperfection,* but it lacks the directness and simplicity of other characteristics. A complete set of characteristics is needed for an adequate picture of the separation. Such a list might include the following: actual yield; theoretical yield; yield error; efficiency (organic); ash error; separating gravity, at actual yield; separating gravity, at actual ash content; separating gravity, from the error curve; probable error of separation; and imperfection.

The calculations involved are presented in a standardized form by means of a schedule*, Table I,

* The schedule is a revised form of a table originally introduced at the Staatsmijnen, Holland.

and two graphs, Figs. 1 and 2, showing the washability curves and the error curve, respectively.

Normally test work needed for evaluating the performance of a cleaner includes a float-sink test from 5 to 8 gravity cuts for the clean coal and the refuse. The cuts may range from 1.25 to 2.5 sp gr, including one separate cut of the feed sample, at 1.60 sp gr.

After being dried and weighed, all the gravity fractions are analyzed for ash. The observational data, including 14 to 20 weight percentages and the same number of ash figures, are now entered in columns A, B, C, and D of the table.

The *actual yield,* Y, is found from the formula

$$Y = 100\,\frac{r-f}{r-c}\ \text{pct} \qquad [1]$$

Generally, the average of two estimates of the yield is calculated, one derived from the ash contents and one from the float percentage at 1.60 sp gr.**

** There is no objection to using a density cut different from 1.60 sp gr, provided it is the same for each of the products concerned.

Thus, r = ash of total refuse, f = ash of total feed, and c = ash of clean coal.

When the formula is used for the second estimate of the yield, r = floats of refuse at 1.60 sp gr, wt pct; f = floats of feed at 1.60 sp gr, wt pct; and c = floats of clean coal at 1.60 sp gr, wt pct. The yield figure is marked down in the outlined part at the foot of column E, and its complement, 100 — Y, in column F. The table is then filled as indicated in the headings of the columns.

The washability curves, Fig. 1, are found from the table, columns L, O, and S being used in conjunction with the cumulative weight percentages given in column M. The error curve, No. 1 in Fig. 2, is found from column T. It signifies the sharpness of separation and provides the estimates of the separating gravity, d; the probable error, r, at separating gravity, d; and the imperfection, I.

The cleaning characteristics are then derived from the curves and entered in the righthand top corner of the table. It is well to check figures found from the curves with the data on the table, by interpolation.

The *theoretical yield* is found from the washability curve of the floats, i.e., curve No. 1 in Fig. 1. It is the yield value that corresponds with the actual ash content of the clean coal. The figure is also calculated from columns M and O, by interpolation.

The *yield error* is the difference between *actual yield* and *theoretical yield.*

$$\text{The } \textit{organic efficiency} = 100\,\frac{\text{actual yield}}{\text{theoretical yield}}$$

The *ash error* is defined as the difference between actual ash content of the clean coal and the ash content that corresponds with the actual yield, on the washability curve of the floats, see curve No. 1, Fig. 1. The latter ash content is called the *theoretical ash content.* This figure is also calculated from columns M and O, by interpolation.

Separating gravity at actual yield is the specific gravity found from the washability curve representing the specific gravity distribution, see curve No. 4, Fig. 1. It is the specific gravity that corresponds with the actual yield; it is also calculated from column M, by interpolation.

Separating gravity at actual ash is found from the same curve. It is the specific gravity of separation that corresponds with the actual ash content and therefore corresponds with the theoretical yield found from curve No. 1 of Fig. 1. The figure is also found from column O, by interpolation.

The separating gravity, d, from the error curve, corresponds with the specific gravity of those particles in the feed of which 50 pct goes with the clean coal during separation and the other 50 pct with the refuse. In curves 1 and 2, Fig. 2, this separating gravity therefore corresponds with a refuse percentage of 50.

The probable error of separation, r, from the error curve, is the specific gravity interval that corresponds with the interval of 25 to 75 in refuse percentage, divided by 2, see curves 1 and 2 of Fig. 2. The refuse percentages per specific gravity interval are also called *Tromp numbers,* or *distribution percentages.*

The imperfection, I, is found from the formula

$$I = \frac{r}{d-t} \qquad [2]$$

where r = probable error of separation, from the error curve; d = separating gravity, from the error curve; and t = density of the medium.

There are a number of other characteristics for expressing the performance of a cleaner, but they will not be defined here. All can be derived from the data presented in the schedule for the calculation of washery results, Table I.

The testing method outlined above is laborious, however satisfactory the information, and simplification of laboratory work would render it more suitable for plant practice. By using the procedure described below it is possible to obtain a very close approximation of cleaner performance from the usual number of float-sink tests without the necessity of conducting fractional ash determinations.

The relationship between the ash content, the volatile matter content, and the specific gravity of coal has been studied by several investigators.[1, 2] It appears that the fractional ash contents and specific gravity of the individual coal are closely related. As specific gravity determination is more simple and less time-consuming than ash determination, various methods have been devised to determine the ash content of a coal from its specific gravity.

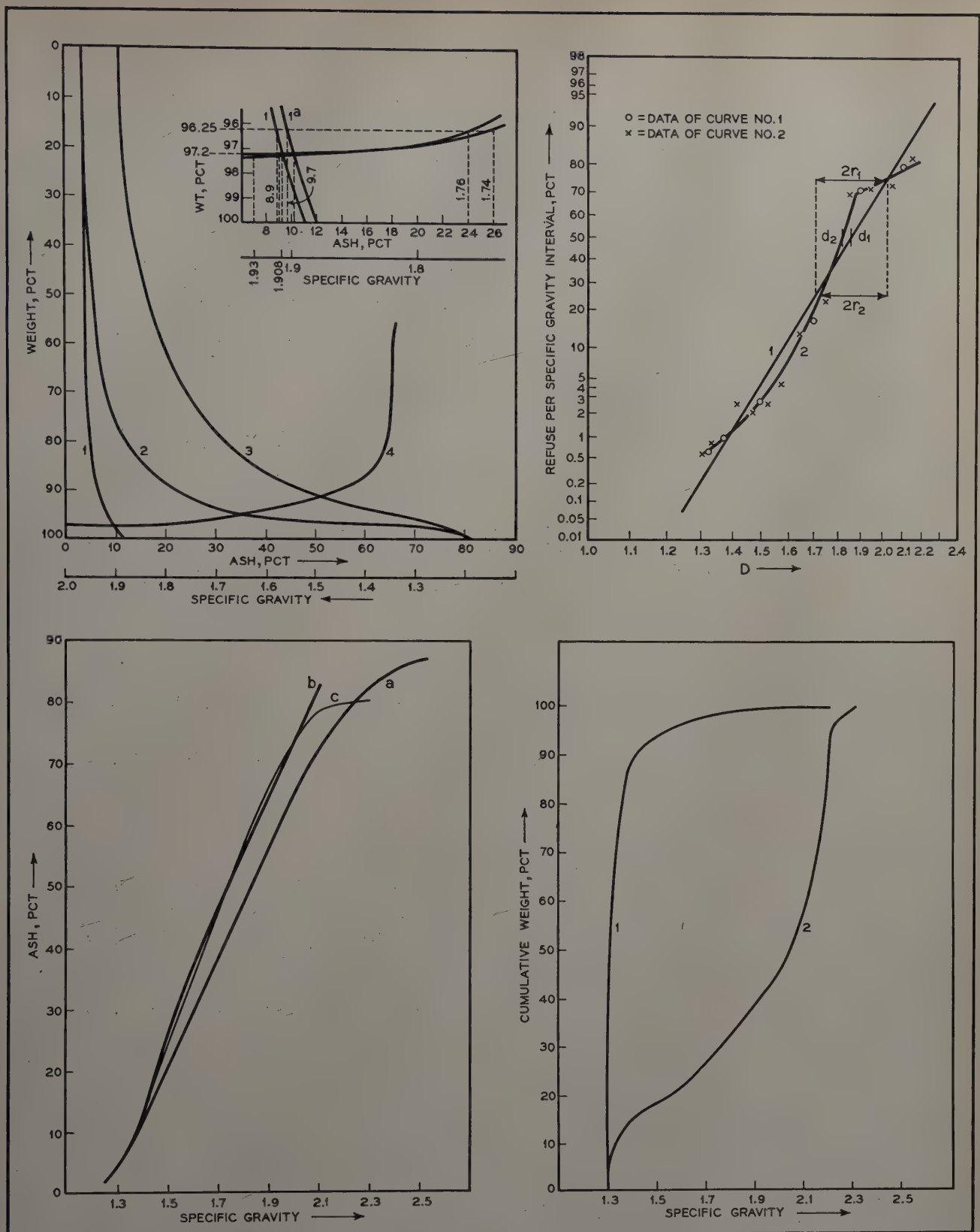


Fig. 1 (upper left)—Curve 1, washability curve of the floats. Curve 2, washability curve of the feed. Curve 3, washability curve of the sinks. Curve 4, specific gravity distribution. Curve 1a (see inset), washability curve of floats for normalized ash contents. Fig. 2 (upper right)—Error curves. Fig. 3 (lower left)—Normalized ash contents. Curve *a*, average curve of bituminous coals. Curve *b*, average curve of subbituminous coals. Curve *c*, average curve of one individual subbituminous coal. Fig. 4 (lower right)—Cumulative weight-density curves: 1, clean coal and 2, refuse.

One possible way would be to find the specific gravity of a sample coal by weighing it in air and then weighing it submerged in water. The corresponding ash content can then be found from the ash-density curve† of that coal. The accuracy of this method, however, is low, one of the reasons being that the ash figure is based on only one observation of the specific gravity.

† This is a short name for the curve representing the relationship between the ash content and specific gravity of a coal.

Table I. Schedule for Calculation of Washery Results

General information

Mine:
Washer: Pneumatic Table
Test No.:
Date:
Test performed by:
Computed by:
Remarks:

Rank (A.S.T.M.): Sub-bituminous B
Grade (A.S.T.M.): A14-F20-S0.7
Size of feed: 1 x 2 in.
Feed, tons per hr.: 5

Washing medium: Air
Density..... t = 0
Viscosity.....

Test data

Duration of test 2.40 - 3.40 p.m.
Weight of Samples Feed 233, clean 90, refuse 89 lbs
Number of increments 80
Feed[1]
Ash content 11.2%
Floats at 1.60 spec. gravity 94.3%
Yield of clean coal, computed from,
(1) the ash contents 96.1%
(2) the floats at 1.60 sp. gr. 96.4%
Mean 96.25%

Test results

Theoretical yield 97.2 %
Yield error 0.9 %
Organic efficiency 99.0 %
Ash error 0.4 %
Separating gravity,
(1) at the actual yield[2] 1.76
(2) at the actual ash content[2] 1.91
(3) from error curve d = 1.86
Probable error of separation[3] r = 0.16

Imperfection $I = \frac{r}{d-t} = 0.09$

SPECIFIC GRAVITY INTERVAL	CLEAN COAL		REFUSE		COMPUTATION OF FEED							CUMUL. SPECIFIC GRAVITY INTERVAL	COMPUTATION OF WASHABILITY CURVES						Error curve[4]
	A	B	C	D	E	F	G	H	I	K	L		M	N	O	P	R	S	T
	Weight %	Ash %	Weight %	Ash %	Weight of clean coal, in % of feed = Yield x A	Weight of refuse, in % of feed = C (100 − Yield)	Ash in clean coal in % of feed = E x B / 100	Ash in refuse, in % of feed = F x D / 100	Ash in clean coal + ash in refuse, in % of feed = G + H	Weight of clean coal + refuse in % of feed = E + F	Ash in feed (washabil. curve No. 2) = 100 I / K		Cumul. weight of feed = K Cumul.	Cumul. ash, in % of feed = I Cumul.	Cumul. ash of floats (washabil. curve No. 1) = 100 N / M	= 100—M	= (Sum I) − N	Cumul. ash of sinks (washabil. curve No. 3) = 100 R / P	Refuse per spec. gr. interval, in % (Tromp numbers) = 100 F / K
- 1.30	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1.30 - 1.35	74.4	5.7	11.9	6.0	71.610	0.446	4.082	0.027	4.109	72.056	5.7	- 1.35	72.056	4.109	5.702	27.944	7.055	25.2	0.6
1.35 - 1.40	15.1	11.5	3.7	10.0	14.534	0.139	1.671	0.014	1.685	14.673	11.5	- 1.40	86.729	5.794	6.680	13.271	5.370	40.5	0.9
1.40 - 1.60	7.5	23.7	5.4	27.8	7.219	0.202	1.711	0.056	1.767	7.421	23.8	- 1.60	94.150	7.561	8.031	5.850	3.603	61.6	2.7
1.60 - 1.80	2.3	44.0	11.8	50.4	2.214	0.442	0.974	0.223	1.197	2.656	45.1	- 1.80	96.806	8.758	9.047	3.194	2.406	75.3	16.6
1.80 - 2.00	0.2	64.4	12.4	68.5	0.192	0.465	0.124	0.319	0.443	0.657	67.4	- 2.00	97.463	9.201	9.440	2.537	1.963	77.4	70.8
2.00 - 2.20	0.5	74.8	50.0	77.7	0.481	1.875	0.360	1.457	1.817	2.356	77.1	- 2.20	99.820	11.018	11.038	0.180	0.146	81.0	79.6
+2.20	-	-	4.8	81.0	-	0.180	-	0.146	0.146	0.180	81.0	-	100.000	11.164	11.164	-	-	-	-
	100.0	9.27	100.0	59.76	96.250	3.750	8.922	2.096	11.164										

FOOTNOTES—[1] The feed figures are used for the computation of the yield, together with those of the clean coal and refuse.
[2] The separating gravities at actual yield and at actual ash are found from washability curves Nos. 1 and 4.
[3] The probable error of separation is one-half of the specific gravity interval lying between Tromp numbers 25 and 75 on the error curve.
[4] The calculation of the error curve (data given in column T) requires the data in columns A, C, E, F, and K only.

Table II. Schedule for Calculation of Washery Results

General information

Mine:
Washer: Pneumatic Table
Test No.:
Date:
Test performed by:
Computed by:
Remarks:

Rank (A.S.T.M.): Subbituminous B
Grade (A.S.T.M.): A14-F20-S0.7
Size of feed: 1 x 2 in.
Feed, tons per hr.: 5

Washing medium: Air
Density..... t = 0
Viscosity..... --

Test data

Duration of test 2.40-3.40 p.m.
Weight of Samples Feed 233, clean 90, refuse 89 lb.
Number of increments 80
Feed[1]
Ash content 11.2 %
Floats at 1.60 spec. gravity 94.3 %
Yield of clean coal, computed from,
(1) the ash contents 96.1 %
(2) the floats at 1.60 sp. gr 96.4 %
Mean 96.25%

Test results

Theoretical yield 97.2 %
Yield error 1.0 %
Organic efficiency 99.0 %
Ash error 0.5 %
Separating gravity,
(1) at the actual yield[2] 1.74
(2) at the actual ash content[2] 1.93
(3) from error curve d = 1.83
Probable error of separation[3] r = 0.14

Imperfection $I = \frac{r}{d-t} = 0.08$

SPECIFIC GRAVITY INTERVAL	CLEAN COAL		REFUSE		COMPUTATION OF FEED							CUMUL. SPECIFIC GRAVITY INTERVAL	COMPUTATION OF WASHABILITY CURVES						Error curve[4]
	A	B	C	D	E	F	G	H	I	K	L		M	N	O	P	R	S	T
	Weight %	Ash %	Weight %	Ash %	Weight of clean coal, in % of feed = Yield x A	Weight of refuse, in % of feed = C (100 — Yield)	Ash in clean coal in % of feed = $\frac{E \times B}{100}$	Ash in refuse, in % of feed = $\frac{F \times D}{100}$	Ash in clean coal + ash in refuse, in % of feed = G + H	Weight of clean coal + refuse in % of feed = E + F	Ash in feed (washabil. curve No. 2) = $\frac{100\ I}{K}$		Cumul. weight of feed = K Cumul.	Cumul. ash, in % of feed = I Cumul.	Cumul. ash of floats (washabil. curve No. 1) = $\frac{100\ N}{M}$	= 100—M	= (Sum I) —N	Cumul. ash of sinks (washabil. curve No. 3) = $\frac{100\ R}{P}$	Refuse per spec. gr. interval, in % (Tromp numbers) = $\frac{100\ F}{K}$
- 1.32	58.0	5.6	8.7	5.6	55.825	0.326	3.126	0.018	3.144	56.151	5.599	- 1.32	56.151	3.144	5.599	43.849	8.935	20.4	0.6
1.32 - 1.35	16.4	8.0	3.2	8.0	15.785	0.120	1.262	0.010	1.272	15.905	7.997	- 1.35	72.056	4.416	6.128	27.944	7.663	27.4	0.8
1.35 - 1.40	15.1	13.2	3.7	13.2	14.534	0.139	1.918	0.018	1.936	14.673	13.194	- 1.40	86.729	6.352	7.324	13.271	5.727	43.1	1.0
1.40 - 1.45	2.7	18.8	1.8	18.8	2.599	0.068	0.488	0.012	0.500	2.667	18.747	- 1.45	89.396	6.852	7.665	10.604	5.227	49.3	2.5
1.45 - 1.50	2.0	24.4	1.1	24.4	1.925	0.041	0.470	0.010	0.480	1.966	24.415	- 1.50	91.362	7.332	8.025	8.638	4.747	55.0	2.1
1.50 - 1.55	1.5	30.4	1.0	30.4	1.444	0.038	0.438	0.012	0.450	1.482	30.364	- 1.55	92.844	7.782	8.382	7.156	4.297	60.0	2.6
1.55 - 1.60	1.3	36.0	1.5	36.0	1.251	0.056	0.450	0.020	0.470	1.307	35.960	- 1.60	94.151	8.252	8.765	5.849	3.827	65.4	4.3
1.60 - 1.70	1.5	46.8	5.7	46.8	1.444	0.214	0.676	0.100	0.776	1.658	46.803	- 1.70	95.809	9.028	9.423	4.191	3.051	72.8	12.9
1.70 - 1.80	0.8	57.2	6.1	57.2	0.770	0.229	0.440	0.131	0.571	0.999	57.157	- 1.80	96.808	9.599	9.916	3.192	2.480	77.7	22.9
1.80 - 1.90	0.1	66.4	5.9	66.4	0.096	0.221	0.064	0.146	0.210	0.317	66.246	- 1.90	97.125	9.809	10.099	2.875	2.270	79.0	69.7
1.90 - 2.00	0.1	74.0	6.5	74.0	0.096	0.244	0.071	0.180	0.251	0.340	73.824	- 2.00	97.465	10.060	10.322	2.535	2.019	79.6	71.8
2.00 - 210	0.2	78.8	14.1	78.8	0.192	0.528	0.151	0.416	0.567	0.720	78.750	- 2.10	98.185	10.627	10.823	1.815	1.452	80.0	73.3
2.10 - 2.20	0.3	80.0	35.9	80.0	0.289	1.346	0.231	1.076	1.307	1.635	79.938	- 2.20	99.820	11.934	11.956	0.180	0.145	80.6	82.3
2.20 - 2.30	-	-	4.8	80.6	-	0.180	-	0.145	0.145	0.180	80.556	- 2.30	100.000	12.079	12.079	-	-	-	-
	100.0	10.17	100.0	61.28	96.250	3.750	9.785	2.294	12.079										

FOOTNOTES—[1] The feed figures are used for the computation of the yield, together with those of the clean coal and refuse.
[2] The separating gravities at actual yield and at actual ash are found from washability curves Nos. 1 and 4.
[3] The probable error of separation is one-half of the specific gravity interval lying between Tromp numbers 25 and 75 on the error curve.
[4] The calculation of the error curve (data given in column T) requires the data in columns A, C, E, F, and K only.

If, on the other hand, the estimate is based on a number of specific gravities, as for the cleaning characteristics subsequently to be described, the accuracy can be improved considerably. In many instances, therefore, it is unnecessary to determine the fractional ash contents once the ash-density curve of a coal is known. The ash figures thus found are termed *normalized ash contents.*

Analyses of coal carried out by the Division of Fuels, Ottawa, during the past 35 years provide much information on the ash-density relationship of Canadian coals. The curves presented in Fig. 3 were derived from 90 float-sink tests and a series of pycnometric tests for the fraction above 1.60 sp gr. Curve *a* represents the average ash-density relationship for bituminous coals. Curve *b* stands for the subbituminous coals. Sufficient data are not available for a curve of the anthracite coals.

These curves can by no means be regarded as typical for an individual bituminous or subbituminous coal. The normalized ash contents must be determined separately for every case. Comparison of such an individual curve with the average curve will then show the difference between the average and the coal in question and will point to its specific cleaning properties. It will appear that the upper part of any individual ash-density curve may digress appreciably from the average because of the different nature of the impurities. Note, for example, curve *c* of Fig. 3. Composition of the coal substance, on the other hand, shows a marked resemblance to other coals of the same rank. The lower part of the individual ash-density curve runs generally parallel to the average, the distance between the two being determined by the difference in volatile matter.‡

‡ Another characteristic of raw coal is that its ash-consist curve is of a binomial frequency curve type. This can be shown by plotting on log-probability paper the free ash content (equals total ash minus inherent ash of lightest fraction) of every specific gravity fraction against the cumulative weight. The resulting curve approximates a straight line, up to around 60 pct ash, where the presence of the impurities becomes predominant. Similar curves have been observed by H. J. de Wijs for the distribution of the metal content in ores.[3] The binomial character of the curve relating to coal, although of no immediate advantage for the calculation of cleaner performance, reveals another statistical property which, conversely, can be retraced from limited information by statistical inference.

Evaluation of Cleaner Performance

The 1x2-in. subbituminous coal analyzed in Table I by the conventional procedure is here examined by the simplified method of normalized ash contents and the results set forth in Table II. Data for the construction of curve *c* of Fig. 3 were derived from a series of four float-sink tests and ash analyses. Note that the main portion of the curve closely follows the average curve for subbituminous coals. The top part deviates distinctly because the impurities consist of a light bentonitic shale containing about 16 pct moisture.

The individual float-sink test was made in the customary way, with 7 to 10 gravity cuts. The relative data of Table I will be used as an example, that is, the figures of columns A and C and the yield figure given at the bottom of column E.§

§ Computation of the yield, as previously described (see Eq. 1) includes finding the float percent at 1.60 sp gr and the total ash content of the feed, clean coal, and refuse, respectively.

Computation of the cleaning characteristics is started by plotting on a separate graph the specific gravities of the clean coal and the refuse against their cumulative weights, see Fig. 4. The curves are used for finding the distribution of material within the specific gravity intervals. Differences in distribution are at once apparent when the ash contents of corresponding specific gravity intervals of the clean coal and refuse of Table I are compared. Ash contents of the refuse cuts are generally higher than the corresponding ash contents of the clean coal. It is clear that these ash contents should be the same if the specific gravity intervals were taken very small. A large number of narrow specific gravity intervals can be derived from the curves, Fig. 4, by interpolation. The resulting data are given in Table II, columns A and C. The normalized ash contents corresponding with the middle of each interval are then found from the ash-density curve *c* of Fig. 3 and filled out in columns B and D of Table II. From here on computation of results is exactly as explained in the previous example and in the table headings.

Comparison of the Standard and Simplified Methods

The two tables, which represent the same separation, can now be compared. The washability curves show certain differences, as expected, but the cleaning characteristics correspond satisfactorily, as shown in Table III.

Table III. Comparison of Data Obtained by Standard and Simplified Methods

Characteristic	Actual Ash Contents, Pct	Normalized Ash Contents, Pct
Theoretical yield	97.2	97.2
Yield error	0.9	1.0
Efficiency	99.0	99.0
Ash error	0.4	0.5
Separating gravity at actual yield	1.76	1.74
Separating gravity at actual ash	1.91	1.93
Separating gravity from error curve	1.86	1.83
Probable error of separation	0.16	0.14
Imperfection	0.09	0.08

The straight line of Fig. 2, curve No. 1, indicates the paucity of data. It shows that a flattering picture of the performance of the cleaner may arise through insufficient information. On the other hand, the method of normalized ash contents, providing more data, defines the shape of the error curve in greater detail. For instance, curve 2 of Fig. 2 is based on 13 points and reveals that in reality the error curve is nonlinear. The shape of the error curve is of particular interest because it aids in detecting certain deficiencies of a cleaner, among others those caused by maladjustment and variations of the separating gravity.[4-6]

Acknowledgment

Permission to publish this paper has been granted by the Deputy Minister, Dept. of Mines and Technical Surveys, Ottawa, Ont.

References

[1] E. S. Grumell and A. C. Dunningham: Distribution of Ash in British Coals. *Colliery Guardian* (1947) **174**, p. 39.

[2] M. J. Berteloot: l'Équispissement des Charbons. *Révue de l'Industrie Minerale* (1946) p. 433.

[3] H. J. de Wijs: Statistics of Ore Distribution. *Geologie en Mijnbouw* (1951) **13**, pp. 365-375; (1952) **14**, pp. 12-24.

[4] Belugou and Vitaux: The Control of the Washing of Fine Coals in the Coalfields. Cerchar Conference, Paris 1950, Paper C 1.

[5] J. Visman: The Status of Coal Cleaning in Western Canada. Bulletin CIMM (January 1954) pp. 15-23; *Trans.* CIMM (1954) **57**, pp. 13-21.

[6] J. Visman: Quality Control Principles in Coal Preparation. Bulletin, CIMM (December 1952) pp. 722-727; *Trans.* CIMM (1952) **55**, pp. 406-411.

Development and Construction Of A Ground-Water Supply

by Owen F. Jensen, Jr.

- Geology
- Hydrology
- Chemistry
- Drilling
- Designing
- Recording

CONSTRUCTION of a ground-water supply includes many operations, which do not end with completion of facilities. Evaluations must be made of the quality of water in various areas and the history of production. Following an analysis of geologic, hydrologic, and chemical data there must be an exploratory drilling program and a study of resulting information. When the well is completed a continuous survey of operating records and basic data is necessary, since designs for future wells are varied as more data becomes available for those already completed.

As the following discussion must be limited to a single undertaking, the customary procedure will be outlined for development and construction of a ground-water supply in which more than three wells are involved. Parts of this procedure are applicable for all ground-water supplies.

Three possible sites were selected for construction of a paper mill. The problem was that the industry daily required approximately 15 million gallons of water of the best quality available; however, if water of the best quality were not available in the desired amount, water of a poorer quality could be used up to half the required amount, about 7 million gallons.

In the preliminary survey approximately 1500 to 2000 square miles were covered for each site. Collected data consisted of several hundred electric logs of oil and gas wells; reports by the U. S. Geological Survey on ground-water resources of localities in and near the area; unpublished records of several hundred water wells including chemical analyses of the water produced and drillers' logs of the wells; records of periodic water-level measurements made in observation wells by the USGS; topographic maps drawn up by USGS and by army engineers; and highway and county road maps. A preliminary report based on study and evaluation of these data was presented to the client, recommending the site with the most favorable and economical ground-water conditions. The study indicated that water of the quality and quantity desired could be developed. In this particular area there were indications that the aquifer could be separated into two zones containing water of different chemical character. This chemical character varied somewhat with area, but principally with depth, a factor which later proved an economic advantage in design of the well field.

After studying the preliminary report, the client weighed other economic factors as well as ground-water advantages and selected a plant site. Authority was given to proceed with detailed study of the chosen area.

Evaluation of preliminary data concerning the area indicated that it should be supplemented with additional information derived from actual exploratory drilling and testing. A flexible plan of exploratory drilling was devised so that data obtained as each test hole was drilled could be correlated with existing data and additional test holes could be located and drilled accordingly. Specific data to be obtained from each test hole were: samples of all formations penetrated; the driller's descriptive log of formations; electric logging surveys; one or more samples of water from selected water-bearing formations; temperature; and water levels and their recovery after periods of production.

The area studied in such detail was approximately 400 square miles. Choice of location for the original test holes was based on the preliminary study. The pattern for these test holes was designed to require a minimum number of holes and to allow for interpolation of data between two, three, and four holes. Geologic, hydrologic, and chemical data gathered after completion of the first four test holes showed that the north section was the most desirable in the area, which was then reduced from the original 400 square miles to about 150 square miles for more detailed and intensive study.

On the basis of findings from the current test a tentative well field was laid out and a drilling program devised. One test hole was located on the actual plant site and later developed into a small pilot production well equipped with a deep well turbine pump. Water from this well was used for general construction requirements.

Another test hole drilled near the pilot production well and adjacent to the proposed route of the well-collection system was used during the pumping test of the pilot production well to determine the degree of interference. From the data thus obtained, calculations were made to determine the coefficients of transmissibility and storage. Three additional holes

O. F. JENSEN, JR., is a Water-Well Engineer with Nicholas A. Rose, Consulting Ground-Water Geologist, Houston, Tex.

Discussion on this paper, TP 39071, may be sent (2 copies) to AIME before Jan. 31, 1955. Manuscript, April 8, 1954. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 1020. The intervening non-Transactions pages appeared in Mining Engineering.

were drilled along the proposed water-collection system. The completed exploratory test-drilling program consisted of eight test holes, five of which, including the pilot production well, were selected for completion into production wells. The remaining three were not completed because of their location relative to final plans for the collection line.

During exploratory testing and drilling the character and texture of the formations were determined by visual examination. Mechanical sieve analyses of samples of water-bearing material gave the grain size, grain-size distribution, uniformity coefficients, and effective sizes. Electric log surveys and drillers' logs indicated depth, thickness, and character of the water-bearing sands. As each water sample was taken from a formation short pumping and recovery tests were made. Together with two pumping and recovery tests made on the pilot production well and the observation well, these gave sufficient data. Variations in temperature and chemical quality of the water at different depths were ascertained; it was found that mineralization increased with depth. The chemical quality of water resulting from the mixing of different waters was calculated. Both specific capacity and drawdown and theoretical drawdown of the pressure level at the pilot production well were determined, and effects on artesian pressure in the observation well due to pumping of the pilot production well were noted. The coefficients of transmissibility and storage at different horizons in the aquifer were recorded.

On the basis of this data a report was prepared for the client. It was stated that the existing water-bearing formation was highly productive and could be depended upon to yield the desired quantity of water at economical levels. Although 60 to 70 million gallons were being withdrawn from this formation daily, it still had ample capacity to produce another 30 to 40 million gallons per day. Quality of the water, even at greater depths, was entirely satisfactory for the requirements outlined.

It was recommended that eight wells be spaced along the collection line at intervals of 2000 ft, seven to operate at 1600 gpm and the eighth to be held in reserve. The wells should be completed at two depths, approximately 750 and 1200 ft, and should alternate in depth. It was calculated that after five years of operation pumping levels would be 200 ft in the 750-ft wells and 350 ft in the 1200-ft wells. It was reported that after five years the levels would stabilize and remain essentially constant unless rate of withdrawal was changed. Natural recharge to the aquifer would be sufficient to supply the 15 million gallons pumped daily, in addition to other known and anticipated withdrawals. Additional areas adjacent to the proposed plant site could be developed to produce water for anticipated requirements.

After the receipt and acceptance of this report the plant site was selected and a final layout made of the well field. Designs for both the shallow and deep production wells included: 1—diameter and length of pump pit; 2—the most economical and efficient pump bowl diameters; 3—diameter and size of conductor casing; 3—type, diameter, size, and length of screen to be installed opposite water-bearing sands; 5—methods for developing the well; and 6—a specific testing procedure to obtain and evaluate hydrologic factors for each well so that preliminary estimates might be amplified and verified. The contract for drilling the production wells was drawn up, bids were taken, and the contract was awarded.

As each well was completed and tested, minor changes were made in the basic design as to gage of screen and size of gravel. Upon completion of each production well pumping tests were made to obtain hydrologic coefficients of the aquifer for the well field. Data from the tests were used to compute future pumping levels originally estimated on data available at the time of the preliminary survey. These hydrologic conditions were considered when pumping equipment was designed. Design, plans, and specifications were drawn and written for the pumping equipment, bids were taken, and a contract was awarded for installation of the deep well turbine pumps.

After all production wells were completed and the deep well turbine pumps were installed, a pumping procedure was set out for a series of production tests of all units before the wells were put into operation for plant processing. Data obtained from the tests and from chemical analysis of water from each well were used to determine the proper production rate for individual wells. On the basis of the testing procedure operating schedules during fluctuations in water demand were also recommended for each well. A rotation plan for using the standby well was outlined.

As in any ground-water development, withdrawals from wells are always accompanied by decline in water level. It is a basic concept that water levels must be depressed to cause water to flow toward the wells. In the well field described in this paper, the predicted decline in water levels meant only an increase in the pumping levels, and not a depletion of the water-bearing sands. However, to determine the accuracy of the predictions, which are based on pumping tests of comparatively short duration, basic data from the wells under normal operation should be collected. These data should include records of total pumpage from the well field, rates of production from individual wells, and pumping levels in the production wells. An analysis of the data in the light of predictions made of future water levels based on the hydrologic factors should be made periodically so the client can be kept informed of current ground-water conditions in the well field. Periodic samples of water from the wells should be collected so that any variation in chemical character of the ground water can be detected.

Unfortunately the described procedure is not yet generally accepted. In many instances specifications, locations, and depth and design of wells have been left entirely to the water-well contractor. Some industries, such as paper mills, mining companies, chemical plants, and power plants, frequently employ the services of consulting hydrologists. On the whole, however, only a small percentage of industries and municipalities have used the techniques of ground-water hydrology in planning and developing their well fields. Furthermore, plans for future expansions of well fields with respect to spacings, individual well yield, and other basic factors are lacking, or only casually considered in the general engineering for the plant. It is only through the application of ground-water hydrology to ground-water supplies that the proper use, development, and conservation of water can be realized.

Acknowledgments

Thanks are expressed to Nicholas A. Rose, who worked with the writer in preparing and supplying basic data for the paper, and to C. K. Hoffmeister, who reviewed and edited parts of the paper.

Structure and Mineralization at Silver Bell, Ariz.

by Kenyon E. Richard and James H. Courtright

SILVER Bell is situated 35 airline miles northwest of Tucson, Ariz., in a small, rugged range rising above the extensive alluvial plains of this desert region. Its geographical relation to other porphyry copper deposits of the Southwest is shown on the inset map in the lower left corner of Fig. 1. The climate is semi-arid. Altitudes range within 2000 and 4000 ft.

Opening of the Boot mine, later known as the Mammoth, in 1865 was the first event of note in the district's history. Oxidized copper ores containing minor silver-lead values were mined from replacement deposits in garnetized limestone and treated in local smelters. Copper production had approached 45 million pounds by 1909 when the disseminated copper possibilities in igneous rocks were recognized. Extensive churn drill exploration carried out during the next three years resulted in partial delineation of two copper sulphide deposits, the Oxide and El Tiro. Although the then submarginal tenor discouraged exploitation of these disseminated deposits, selective mining of orebodies in the sedimentary rocks continued intermittently until 1930, providing a production total of about 100 million pounds of copper.

The American Smelting & Refining Co. began exploratory and check drilling in 1948 and subsequently made plans for mining and milling the Oxide and El Tiro orebodies at the rate of 7500 tons per day. Production began in 1954 at a rate of about 18,000 tons of copper annually.

Formations ranging in age from Pre-Cambrian to Recent are exposed in the Silver Bell vicinity. The more erosion-resistant of these, Paleozoic limestone and Tertiary volcanics, predominate in the scattered peaks and ridges comprising the Silver Bell mountains. The porphyry copper deposits are located along the southwest flank of these mountains in hydrothermally altered igneous rocks. These are principally intrusives which cut Cretaceous and older sediments and are considered to be components of the Laramide Revolution.

For three-fourths of its length the zone of alteration strikes west-northwest, Fig. 1. There now is no single structure that accounts for this alignment. However, indirect evidence suggests that a fault representing a line of profound structural weakness existed in this position prior to the advent of Laramide intrusive activity. This line will be referred to as the *major structure*. It was obliterated by the Laramide intrusive bodies but exerted a degree of control on their emplacement, as evidenced by their shapes and positions. The influence of fault structures on the shapes of intrusives in other porphyry copper districts has been noted by Butler and Wilson[1] and by others.

As shown on the inset map on Fig. 2, a fault of parallel trend and considerable displacement lies to the north. This fault is now marked by a line of small Laramide intrusive bodies. To the south is a third fault of large displacement. Evidence of its age in relation to the Laramide intrusions and mineralization is not recognized, but its conformance in strike with the other two major faults is significant. These three breaks establish a pronounced trend of regional faulting. They are high-angle, and the southerly one may be reverse. Stratigraphic separations on these faults are of the order of several thousand feet.

The local Paleozoic section is about 4000 ft thick. It is composed predominantly of limestone with a basal quartzite member. The Cretaceous section appears to exceed 5000 ft. Conglomerates, red shales, and arkosic sandstones (the youngest) characterize the three principal members.

Intrusion of alaskite marked the beginning of Laramide igneous activity. It was emplaced as an elongate stock with one side closely conforming to the major structure line throughout a distance of nearly 4 miles. The alaskite was at one time regarded as a thrust block of pre-Cambrian rock[1]; however, its intrusive relationship and consequent post-Paleozoic age has been established by inclusions of limestone found in outcrops north of El Tiro.

The next event was the intrusion of a large stock of dacite porphyry into Paleozoic sediments and alaskite. The stock was some 3 miles wide and at least 6 miles long in a northwesterly direction. It was sharply confined along its southwest side by the major structure line. A number of large pendants of moderately folded Paleozoic sediments occur within and along its southwest edge. Thus the inferred, original major fault between Paleozoic and Cretaceous sediments became a contact between alaskite and Paleozoic sediments and then a contact between dacite porphyry and alaskite. Andesite porphyry may have been intruded later than the dacite porphyry, but relationships are not clear; it may be simply a facies of the latter.

The intrusive activity was at this stage interrupted by an interval of erosion. The erosion surface probably was rugged, as there were local accumulations of coarse, angular conglomerate. Subsequently a series of volcanic flows and pyroclastics several thousand feet thick was deposited. A similar unconformity has been recognized elsewhere in the Southwest, particularly in the Patagonia Mountains near the Flux mine some 75 miles southeasterly. Here, as at Silver Bell, volcanics were deposited on an erosion surface cut in Cretaceous and older sediments which had been intruded by alaskite.

Though no evidence is offered that closely defines the age of this unconformity, and proper analysis of the problem is beyond the scope of this paper, it is

K. E. RICHARD and J. H. COURTRIGHT, Members AIME, are Chief Geologist and Geologist, respectively, Western Mining Dept., Southwestern Div., American Smelting & Refining Co., Tuscon, Ariz.

Discussion on this paper, TP 3920I, may be sent (2 copies) to AIME before Jan. 31, 1955. Manuscript, March 1, 1954. New York Meeting, February 1954.

interesting to speculate that it may mark the close of the Cretaceous Period and provide a distinction between Tertiary and Cretaceous igneous rocks within the Laramide Revolution.

Subsequent parallel faulting along the major structure line sliced the volcanics and Cretaceous sediments into horst and graben structures. These faults are remarkably persistent southeasterly, extending several miles beyond the limits shown in the map. It is not clear whether or not they originally extended through the northwest part of the district; they may have terminated against the earlier east-west fault shown on the inset map in Fig. 2. The formation of these faults indicates at that time a still existent deep-seated zone of weakness along the major structure line.

Monzonite stocks and contemporaneous dikes were then emplaced along and near this line, obliterating portions of the faults described in the foregoing paragraph. The stocks are elongate parallel to the major structure line. The dikes are distributed along this line but trend across it, for the most part, with an average east-northeast strike. Systems of close-spaced parallel fractures then developed. Like the dikes, these fractures are distributed along the major structure and strike across it. Alteration and sulphide mineralization then took place. The deposition of sulphides, particularly chalcopyrite, was controlled in detail by the cross-trending fractures. Although these were distributed along the major structure line as a narrow band, it is notable that throughout much of its length there are now no fault sructures to account for this trend.

Post-sulphide dikes of andesite represent the last intrusive activity in the immediate district. Curiously, most of these dikes parallel the strike of the major structure, although it would seem that the cross-breaking fractures represented available lines of weakness. This serves to emphasize the major structure line as being a profound, deep-seated zone of weakness persisting through a long period.

Uplift and erosion of the region during late Tertiary or Quaternary time exposed the lean primary mineralization to processes of weathering and enrichment, resulting in the accumulation of the two chalcocite orebodies.

Small plugs and flows of basaltic lava occur in the flats surrounding the Silver Bell range. These are later in age than the Gila conglomerate and mark one of the more recent events in the geologic history of the region.

Structural Control of Hypogene Mineralization

As in the majority of porphyry copper deposits, the principal primary sulphide minerals at Silver Bell are pyrite and chalcopyrite. Although occurring as disseminated grains, they are more abundant as narrow veins or seams which are usually near-vertical in attitude and persistently parallel. Varying in thickness from paper-thin to several inches and in spacing from inches to several feet, these thin sulphide sheets occur as groups of various sizes throughout the narrow northwest-trending zone of hydrothermal alteration. (Because of the small scale, a single line in the pattern of *Mineralized Fissures* on Fig. 1 usually represents a large number of parallel structures, rather than individual.) In detail the average individual fissure appears as a thin quartz-sulphide seam encased by a uniform band of sericite. The fissures are predominantly oriented in the northeast quadrant; a small proportion strike northwest and a few are random. From a broad viewpoint there are no intersections of consequence among these groups. Within groups changes in strike occur gradually and result in curving trends. As noted earlier, these systems of mineralized fissures are distributed along the major structure line, and it is assumed they were formed in response to deep-seated uniform stress related to this line.

At least a few hundredths of 1 pct copper is present nearly everywhere in the altered zone; better values occur where there are mineralized fissures and the best values where the fissures are close-spaced. The two comparatively large groups of these close-spaced structures coincide with the positions of the two orebodies, Fig. 1. However, the actual structural, mineralogical, and lithological distinctions among these and other, smaller groups are minor, and the factors that controlled the position and size of these two groups are not clearly evident. The strong east-west fault which terminates in the Oxide area may have influenced the concentration of fracturing there, and at El Tiro the sharp bend in the alteration zone and the group of northeast-striking dikes likewise may indicate a cross-trending line of weakness that localized stresses. Nonetheless, the importance of these structural conditions is not clearly demonstrated, and no good evidence is found to explain the structural cause of the more intense fracturing which localized the two orebodies in their present positions in preference to other locations along the major structure line.

Outside the zone of alteration the dacite porphyry is finely fractured and jointed throughout most of its large exposed area. In sharp contrast to the systems of parallel fissures in the alteration zone, these breaks in the dacite porphyry are almost completely disoriented; parallelisms are rare and traceable for only a few inches or feet. They appear to be premineral where they are found in the alteration zone in the westerly and southwesterly portions of the dacite porphyry. Evidently in physical aspect this formation was exceptionally well prepared to be mineralized, perhaps better than the rocks of the ore zone proper. The fact that it was mineralized only to a minor degree may be accounted for by the absence of systematic fractures. That is, only the systems of parallel fractures were connected with the deep-seated source of mineralization, and the pervasive breaking of the dacite porphyry did not alone qualify it for mineralization.

Except the post-mineral andesite dikes, all igneous rocks within the narrow northwest-trending zone shown on Fig. 1 are hydrothermally altered. Variations in the intensity or in the completeness of the process have been subdivided by Kerr[2] into five stages. His analysis demonstrated, among other things, that the known orebodies occur within the more strongly altered areas.

The area outlined on Fig. 1 includes all degrees of alteration, but no differentiation is made. It merely represents the areal extent of bleached-looking, igneous rocks showing evidence in the leached outcrops of pre-existing disseminated sulphides, principally pyrite. The transition to relatively fresh rock is sharp in many places, particularly along the contact with sedimentary rocks and on the faults in the southeast portion. However, along most of the southwest margin the transition is gradational, and the limit is an arbitrary line.

Tactite, composed essentially of garnet, quartz, and lime-silicate minerals, is confined to a narrow belt along the southwest margin of the limestone

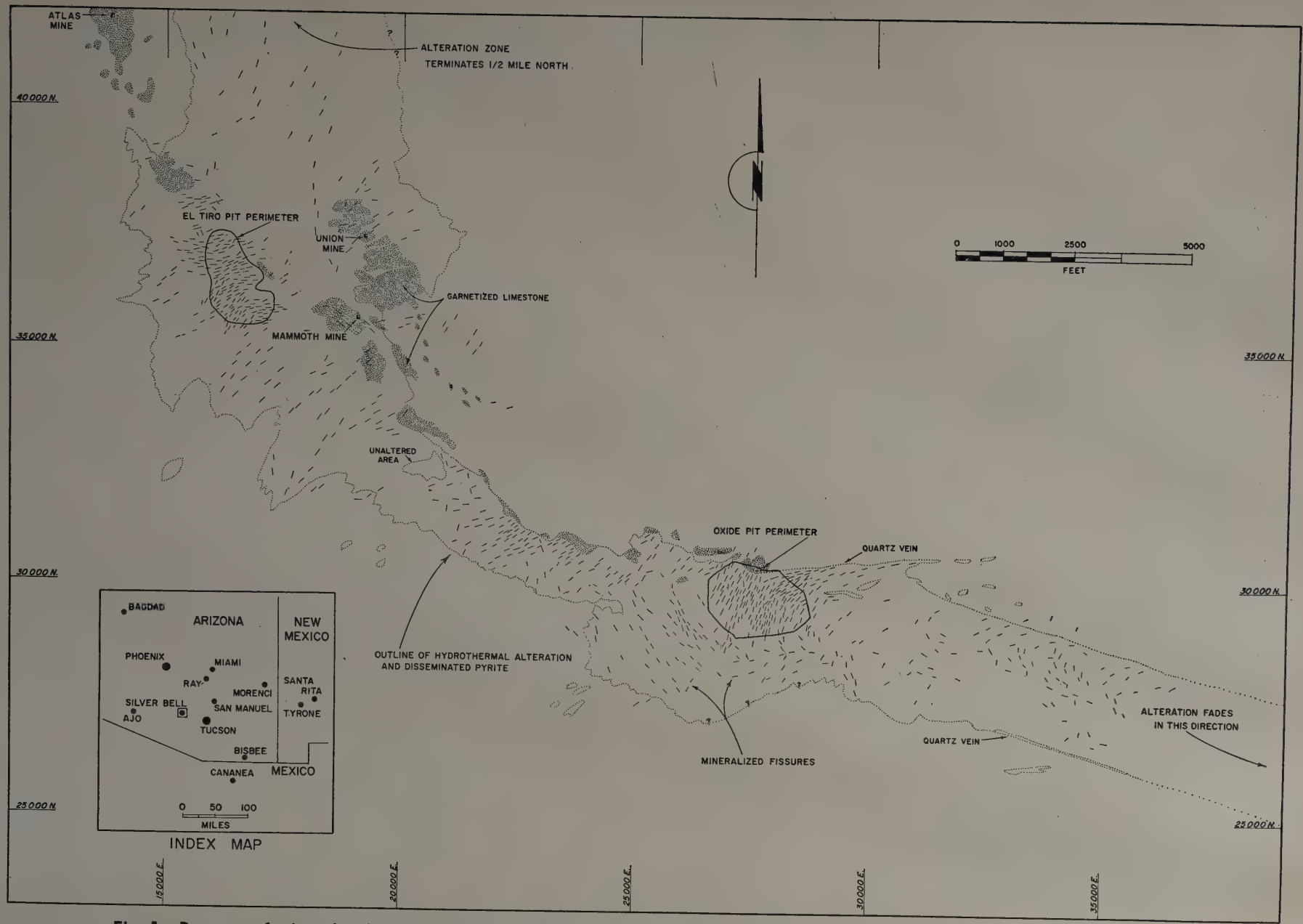


Fig. 1—Patterns of mineralized fissures, zone of hydrothermal alteration, and areas of pyrometasomatism in sedimentary rocks.

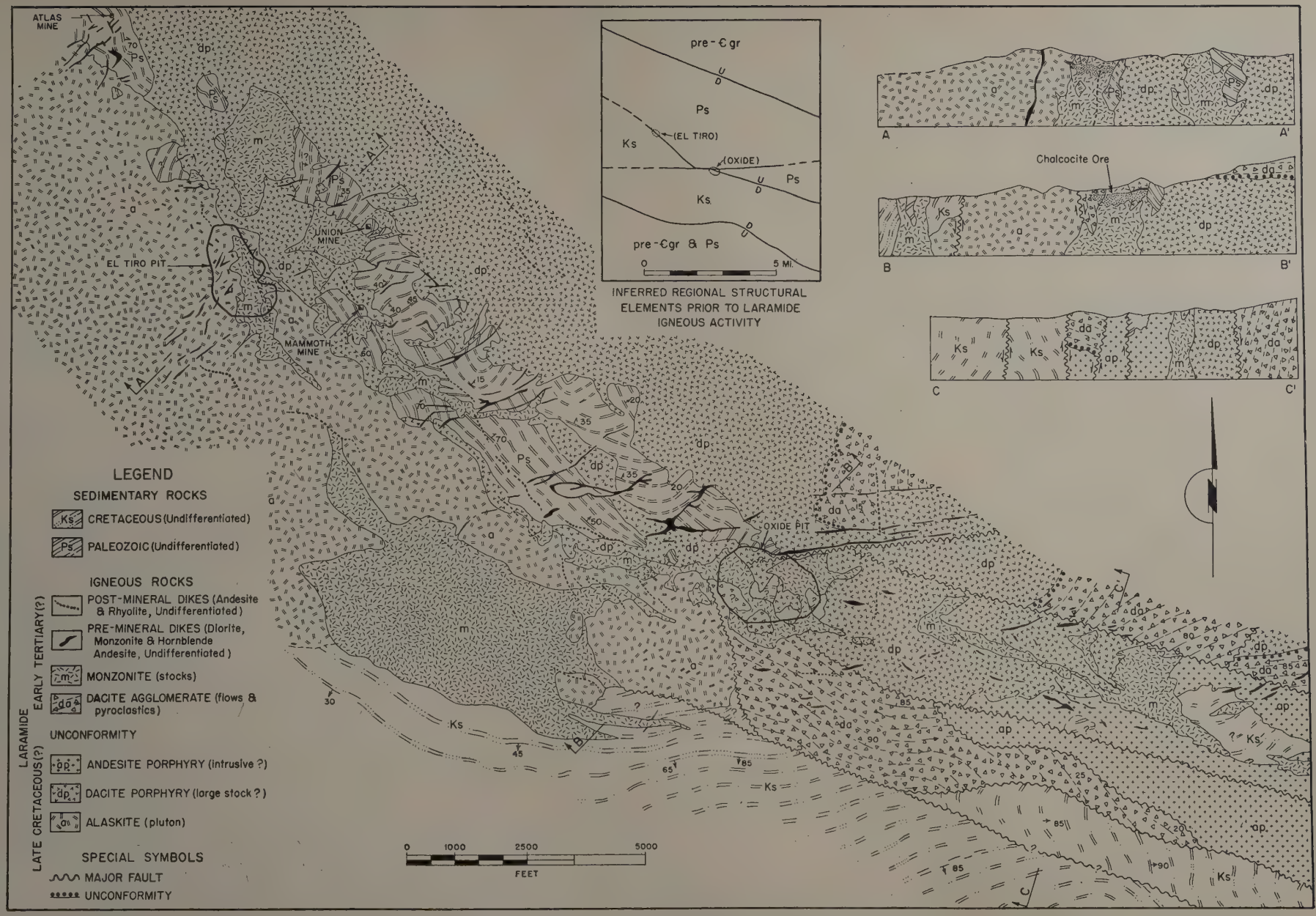


Fig. 2—Geologic map, Silver Bell district, Pima County, Ariz.

pendants, except in the vicinity of the Mammoth and Union mines where it has replaced the full width of the sedimentary block. It has been suggested by Stewart[3] that the dacite porphyry and monzonite are responsible for this *contact metamorphism*. Areal distribution of this tactite is such that, if it were to be considered strictly as a contact phenomenon, the alaskite would be as related to it as the other intrusives. Without going into the problem in detail, it is worth noting that the tactite occurs along the major structure line in such a manner as to indicate a close genetic connection with it. Supporting evidence in the form of well-defined structural controls of individual pods of tactite is not recognized. An occasional mineralized fissure cuts the tactite in the Mammoth and Union deposits, although the primary chalcopyrite orebodies have little obvious structural control. Elsewhere fissures in igneous rocks end abruptly at tactite margins.

Supergene Enrichment

The two ore deposits consist of rudely tabular accumulations of chalcocite from 100 to 200 ft thick. Lying beneath about 100 ft of leached capping, they were formed by two to threefold enrichment of the copper contained in the primary mineralization. Typical ore is composed of altered rock and sulphides in a ratio of about 10 to 1 by weight.

Most of the capping over the orebodies contains less than one-tenth of 1 pct copper as cuprite, or other oxidation products, mingled with the limonite. Occasionally somewhat higher values occur where copper has been precipitated as silicates and carbonates by reactive gangue material present in less altered rock. Within the orebodies, where alteration is strong and the gangue is nonreactive, the upper limit of the sulphide zone (the base of oxidation) appears on opencut faces as a sharply defined, highly irregular line. Only rarely is there a transition zone of mixed sulphide and oxidized minerals. In general shape the base of oxidation conforms to modern topograhy, even though local relief exceeds 200 ft. The water table for the most part now is well below the chalcocite zone.

Some of the irregularities of the base of oxidation are caused by displacement on post-chalcocite faults, but most are due to variations in rock permeability. This is evidenced by the dense siliceous character of a few sulphide remnants occurring well up in the leached zone, and by leached indentations of the sulphide zone along some of the fissures.

It is significant that although the base of oxidation shows general conformance to the topography, in detail it is a highly irregular, sharply defined front. Its present shape may have been produced by modification of a pre-existing base, one that was established during relatively moist climatic periods of the past. Under such conditions the water table would have oscillated at uniformly shallow depths, serving to limit the depth of oxidation and thereby determining the shape of its base to some extent. Otherwise, under conditions involving depression of the water table, the oxidation process apparently proceeded in the vadose zone independent of the water table, advancing downward on a sharply defined front whenever oxygen-charged meteoric water reached it.

Opinions vary both as to the role of the water table in the deposition of chalcocite and the reason for distribution of the chalcocite through a considerable vertical range. At Silver Bell pyrite and preferentially chalcopyrite are only partially replaced by chalcocite immediately below the line at the base of oxidation as well as on down through the zone of enrichment. This condition favors the theory that chalcocite is deposited at or below the water table. That is, the dissolved copper on its downward course bypassed available chalcopyrite and pyrite until it reached the water table where it formed chalcocite. The partial replacement of primary sulphides by chalcocite and its vertical distribution, as now existing, may then be explained as originating through the numerous cyclic fluctuations of the water table position.

Leached Outcrops

In the formation of most disseminated chalcocite deposits the enrichment process takes place progressively: copper is repeatedly dissolved, carried downward, and precipitated. It has been well established by Blanchard,[4] Locke[5] and others that under these conditions limonites of certain colors and textures are left behind in the leached capping as evidence of the pre-existing chalcocite.

The Silver Bell district provides exceptionally good examples of this phenomenon, but limonites of chalcocite derivation are not confined to the outcrops over the orebodies. They are widely dispersed through the zone of alteration. Proper interpretation of their significance in respect to ore possibilities rests mainly on quantitative rather than qualitative appraisal. Mapping of the Silver Bell outcrops on this basis provided a valuable guide in exploration drilling. Results have demonstrated conclusively that the pattern of relatively strong copper mineralization at depth is reflected in the outcrops by the distribution and abundance of diagnostic limonites.

Previous Work and Acknowledgments

The first scientific study of the district was published in 1912 by C. A. Stewart.[3] Considerable field and laboratory work has been done in more recent years by several groups and individuals, including the writers, all reporting privately to the American Smelting & Refining Co. Roland Blanchard conducted leached outcrop studies in a portion of the area. Harrison Schmitt with H. M. Kingsbury and L. P. Entwistle mapped structure and mineralization in the central part of the district. Paul F. Kerr studied the alteration features, and later published a comprehensive paper[2] on the district. Thomas Mitcham mapped structural features in the surrounding area. The writers have drawn considerably on unpublished data, particularly in compilation of the geologic map. The high quality and usefulness of the work of these men is gratefully acknowledged, but unfortunately it is not feasible to give specific individual credits.

Thanks are due the American Smelting & Refining Co. for permission to publish this paper.

References

[1] B. S. Butler and E. D. Wilson: Clifton-Morenci District. Ariz. Bur. Mines *Bull. 145*, p. 74 (1938).

[2] Paul F. Kerr: Alteration Features at Silver Bell, Arizona. GSA *Bull.* (1951) **62**, pp. 457-480.

[3] Charles A. Stewart: The Geology and Ore Deposits of the Silver Bell Mining District, Arizona. *Trans.* AIME (1912) **43**, pp. 240-290.

[4] Roland Blanchard: Interpretation of Leached Outcrops. *Journal of the Chemical, Metallurgical and Mining Society of South Africa* (May 1939).

[5] Augustus Locke: *Leached Outcrops as Guides to Copper Ore.* Williams and Wilkins, Baltimore, 1926.

Discussion*

Contents

A — Metal Mining

Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes. (Paper by W. A. Boyer, *Transactions* AIME, **199**, 989; *Mining Engineering*, October 1954. Discussion by B. E. Grant.) 1101

The Use of Wooden Rock Bolts in the Day Mines. (Paper by Rollin Farmin and Carville E. Sparks, *Transactions* AIME, **196**, 922; *Mining Engineering*, September 1953. Discussion by Edward Thomas.) 1101

An Analysis of Mine Opening Failure by Means of Models. (Paper by Bernard York and John J. Reed, *Transactions* AIME, **196**, 705; *Mining Engineering*, July 1953. Discussion by J. P. Zannaras.) 1102

B — Minerals Beneficiation

Flotation and the Gibbs Adsorption Equation. (Paper by P. L. de Bruyn, J. Th. Overbeek, and R. Schuhmann, Jr., *Transactions* AIME, **199**, 519; *Mining Engineering*, May 1954. Discussions by W. E. Ewers and Masayoshi Wada.) 1103

Manganese Upgrading at Three Kids Mine, Nevada. (Paper by S. J. McCarroll, *Transactions* AIME, **199**, 289; *Mining Engineering*, March 1954. Discussion by J. Bruce Clemmer, J. B. Rosenbaum, and C. H. Schack.) 1105

A Physical Explanation of the Empirical Laws of Comminution. (Paper by D. R. Walker and M. C. Shaw, *Transactions* AIME, **199**, 313; *Mining Engineering*, March 1954. Discussions by Dimitri Kececioglu, George B. Clark, and Fred C. Bond.) 1106

F — Coal

Underground Electrocarbonization of Coal and Related Hydrocarbons. (Paper by T. C. Cheasley, J. D. Forrester, and Erich Sarapuu, *Transactions* AIME, **199**, 908; *Mining Engineering*, September 1954. Discussions by John G. Tripp and James L. Elder.) 1108

Longwall Mining and Mechanization, with Special Reference to Nova Scotia. (Paper by Frank Doxey, *Transactions* AIME, **199**, 701; *Mining Engineering*, July 1954. Discussion by Louis A. Turnbull.) 1109

Cleaning Various Coals in a Drum-Type Dense-Medium Pilot Plant. (Paper by M. R. Geer, W. A. Olds, and H. F. Yancey, *Transactions* AIME, **196**, 696; *Mining Engineering*, July 1953. Discussion by John Griffen.) 1110

Frontiers in Heat Extraction from the Combustion Gases of Coal. (Paper by Elmer R. Kaiser, *Transactions* AIME, **199**, 304; *Mining Engineering*, March 1954. Discussion by G. A. Vissac.) 1112

Exploration of the Oaxaca Coal Fields in Southern Mexico. (Paper by Luis Toron and Salvador Cortes-Obregon, *Transactions* AIME, **199**, 505; *Mining Engineering*, May 1954. Discussion by John D. Price.) 1113

H — Industrial Minerals

Alkali Reactivity of Natural Aggregates in Western United States. (Paper by William Y. Holland and Roger H. Cook, *Transactions* AIME, **196**, 991; *Mining Engineering*, October 1953. Discussions by Dexter H. Reynolds and W. L. Merritt.) 1114

Characteristics of Titaniferous Concentrates. (Paper by L. E. Lynd, H. Sigurdson, C. H. North, and W. W. Anderson, *Transactions* AIME, **199**, 818; *Mining Engineering*, August 1954. Discussion by D. R. Grantham.) 1116

---

* TP 3911

A — Metal Mining

Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes.

by W. A. Boyer

DISCUSSION

B. E. Grant *(U.S. Smelting, Refining & Mining Co., Salt Lake City)*—There are several exceptions from the practical operating viewpoint that might be taken with the paper. The author recommends using a "curve" in plotting safety factors of ropes and suggests that the U. S. Bureau of Mines change its present "step curve" method to the "curve" method. From an engineering theoretical point of view that is possible, but from a practical standpoint it is not possible. Rope manufacturers fabricate their ropes in diameter gradients of 1/16 in. to 1/4 in. which automatically sets up a step pattern gradient. If ropes were manufactured in the diameter gradients of 1/1000 of an inch or thereabouts, the curve method could be used. Also, from a practical standpoint, if an operator is to hoist from a depth of 4000 ft, for example, he will determine the size of the rope necessary for that depth, and it is only natural that he will have safety factor to spare at lesser depths, and in plotting this excess capacity the natural end result is a curve. The U. S. Bureau of Mines tables are set up so that the operator will know what size rope at the required safety factor he will need at his maximum depth. He is not concerned with lesser depths.

W. A. Boyer *(author's reply)*—Mr. Grant states, "Rope manufacturers fabricate their ropes in diameter gradients of 1/16" to 1/4" which automatically sets up a step pattern gradient." There is no connection or relationship between the "size" pattern gradient and the present "factor of safety" step pattern gradient. The inherent factor of safety values for any particular size of rope with a given connected load when plotted for various depths is a *curve*, and with modern rope material and construction the inherent factor of safety curve is quite far removed from the present recommended factor of safety step curve. This is shown quite clearly in Figs. 3 and 4 of my paper.

Mr. Grant says, "If an operator is to hoist from a depth of 4000 ft, for example, he will determine the size of rope necessary for that depth, and it is only natural that he will have safety factor to spare at lesser depths." This is not true. I have prepared the paper with the prime purpose of showing the fallacy in only checking the factor of safety at the lowest depth and then trusting it is all right for the upper levels. I stressed in the paper the difference and showed in Fig. 4 that this difference is on the danger side when hoisting from levels above 4000 ft in the above-mentioned instance. With the installation calculated for a factor of safety of 4 at 4000 ft, static loading, a comparison for the upper levels is as follows:

Item	Depth of Hoisting, Ft				
	4000	2500	1500	750	250
Recommended Bureau of Mines values	4	5	6	7	8
Actual values considering static loading	4	4.53	4.96	5.35	5.64
If an acceleration rate of 3.216 ft per sec per sec is present the actual values would be	3.64	4.11	4.51	4.86	5.12

By far the simplest factor of safety rule would be to require that the factor of safety at 2500 ft equal 5.55. The inherent characteristic curve of the rope would then give a factor of safety at the various depths as indicated by my proposed curve. The characteristic curves for various size rope vary but slightly and for high rates of acceleration would allow a slight increase in factor of safety for the upper levels.

Use of Wooden Rock Bolts in the Day Mines

by Rollin Farmin and Carville Sparks

DISCUSSION

Edward Thomas *(U.S. Bureau of Mines, Washington, D. C.)*—This excellent article on an ingenious and successful installation of wooden rock bolts loses much of its effectiveness through an attempt by the authors to establish the general adaptability of this type of support. Possibly because they have only local experience as a guide, they imply erroneously in the first paragraph that the Bureau of Mines has been opposed to wooden bolting, although later in the article reference is made to a Bureau publication on the subject.

In 1947 the Bureau of Mines decided to sponsor experiments in roof bolting in coal mines because the experience of the St. Joseph Lead Co. had indicated that this type of roof support appeared to be a roof-fall accident-prevention measure that might be adopted and also effect economies in operating cost.

Justification for this belief has been borne out, particularly in coal mining. This program has been carried out under the following general program:

1—The Roof Control Section of the Bureau of Mines has advocated and initiated underground experiments under controlled conditions with many different ideas related to rock bolting since 1947. In no instance has cooperation in developing an idea been refused, no matter how ridiculous it may have seemed at first glance, if it has been requested by a mining company.

2—The Bureau of Mines has no police powers over the selection of roof-support devices and seeks none.

3—At the request of an operator, the Bureau of Mines will make comparative tests on various types of bolts underground and will submit resultant data for the operator's consideration.

4—Wherever possible, the Bureau publicizes successful installations, so that others may benefit from this experience; however, it is careful not to discriminate between manufacturers of bolts or bolt-installation equipment. Unsuccessful experiments probably should also be publicized, but from a practical standpoint it is usually not feasible.

In other words, the role of the Roof Control Section of the Bureau is in line with the original purpose for which the Bureau was first organized: to serve industry through gathering, analyzing, and disseminating factual data. It would seem to be logical that the starting point for any experimentation on roof bolts in coal mines should be the time-proved practice of the

St. Joseph Lead Co. in sedimentary rocks; however, different sets of operating and economic conditions made it desirable and inevitable that many changes would be made during the adoption. At no time has the Bureau resisted these changes. It has exerted influence only to insure that experiments be carried on in a safe and orderly manner.

The first installation of wooden bolts was made in 1949 by Sterling S. Lanier, Jr., in the Rio Verde mine of the Norton Coal Corp., operating in western Kentucky, and the first publicity given this installation was at the 1949 National Safety Council in Chicago by Leon Kelly of the Roof Control Section of the Bureau of Mines, who had assisted Lanier in his experiments. Several coal mines in central Pennsylvania, northern West Virginia, and western Kentucky became interested in the Lanier bolt and have done considerable experimental and development work. It was not successful and interest lagged for a time. In 1952 G. R. Spindler of the Mining Dept., in cooperation with the Forest Products Laboratories of West Virginia University, started a test program in the Scotts Run field of northern West Virginia with a wooden bolt of new design. It is understood that the results of these tests are not yet ready for publication.

The Boone County Coal Corp., in cooperation with the Bureau of Mines, has been experimenting with wooden bolts in the Winding Gulf field of central West Virginia since 1949 and has not yet developed one that meets with the company's entire satisfaction, but a new wooden bolt and a new installation technique still offer promise of success, if they can be brought within a competitive cost range.

Therefore, contrary to the impression the authors seem to have, more widespread use of wooden bolts is not being and has not been hindered by opposition of the Bureau of Mines but through the inherent limitations imposed on this method by the materials and the techniques developed. Regardless of any state or federal governmental influence, considering the highly competitive coal market, anyone is indeed näive who thinks that a saving of $1.05 in the material cost of a bolt would not swing at least three-fourths of the coal mines to wooden bolting if it could be done safely.

It is believed that there is a definite place for wooden bolts in mining, and the article illustrates the ideal application: to reinforce the skin area of an arched opening and thus maintain the integrity of the arch by preventing sloughing of loose material through a maximum of bearing surface provided by the slabwood, and through the large amount of bearing surface between the wooden rod and the sides of the hole which provides shear strength to stiffen the structure. In such a formation, that is wet, loosely consolidated quartzite, application of tension at the bearing plate is unnecessary, and it would be impossible to maintain such tension if it were necessary.

Proponents of wooden bolts for beam building point out that many woods are comparable to steel in tensile strength parallel to the grain. However, no one has as yet suggested a practical way to take advantage of this strength. Pull tests with hydraulic jacks on such bolts show that only a small fraction of this strength can be utilized. Actually in the way these bolts are used, this test is not a criterion of effectiveness. If a wooden bolt is installed to maximum efficiency its maximum usefulness is in shear, not in compressing the rock by applying tension between a bearing plate and a fastening device inserted in the drilled hole, and wood can not resist shear efficiently, especially parallel to the grain. It is suggested that the back of the hole can be reamed, and by driving a wooden pin with a ram or a percussion tool obtain a much better bearing at the back than has been possible heretofore. This may well be so, but no practical bearing plate has been suggested that will take full advantage of this increased anchorage.

From investigative data gathered by the Bureau of Mines the following limitations prevail:

1—Wooden bolts have not been successful to date in compacting rocks in significant spans, except in marginal support cases, where approximately 90 pct of the rock would stand intact without any form of artificial support. The installation described in I. C. 7637 is of this nature.

2—It is unsafe to depend on the swelling action of the wood in a humid, wet atmosphere to expand the wood in the drilled hole, unless it is established that there is no seasonal humidity change in the mine. This is not a factor at the Day Mines.

3—The cost of drilling the large-diameter holes, plus the cost of adequate storage underground and on the surface, increased installation time, and other factors that are not immediately apparent, sometimes increase the installed cost of wooden bolts beyond the price of installed steel bolts.

An Analysis of Mine Opening Failure by Means of Models

by Bernard York and John J. Reed

DISCUSSION

J. P. Zannaras *(U.S. Tungsten Corp., Congress, Ariz.)* —In the investigation described in the paper by Bernard York and John J. Reed, the 8x8x8-in. square block was subjected to compressive forces from three sides, top, side, and rear.

Let us refer to the stresses set up by these forces as S_z due to top pressure, S_y due to side pressure, and S_x due to rear pressure.

At the section across the 2-in. hole (when failure occurred at 317 psi) the compressive stress S_y was equal to $317 \times 8/6 = -422$ psi, the top compressive stress S_z is of the same intensity, —422 psi, and the rear compressive stress S_x equals —317 psi.

The true stress is $T = S_y - \lambda S_z - \lambda S_x$.

Taking λ as 0.2,*

$$T = -422 + 0.2 \times 422 + 0.2 \times 317 = -274 \text{ psi.}$$

* See Mansfield Merriman's *Mechanics of Materials*, 11th ed., pp. 360 and 361.

Taking the case when the opening was ½ in. and failure occurred at 517 psi,

$$S_y = 517 \times \frac{8}{7.5} = -537 \text{ psi}$$

$$S_z = 517 \times \frac{8}{7.5} = -537 \text{ psi}$$

$$S_x = -517 \text{ psi}$$

The true stress $T = S_y - \lambda S_z - \lambda S_x$

$$T = -537 + 0.2 \times 537 + 0.2 \times 517 = -327 \text{ psi.}$$

It can be shown that the above fundamental formula holds good not only for the above two examples but for all sizes of openings used, because when this formula is expressed in terms of the external pressure and size of the opening it agrees perfectly with the experimental results obtained by the authors and given in Fig. 12 of their paper.

General formula: $T = S_y - \lambda S_z - \lambda S_x$.

Since all stresses are compressive the formula can be written for this particular case as follows:

$$T = -S_y + \lambda S_z + \lambda S_x \qquad \text{since } S_y = S_z$$

$$T = S_y(\lambda - 1) + \lambda S_x$$

$$S_y = \frac{P \times 8}{8 - V} = S_z$$

$$S_x = P$$

P = pressure, psi

V = size of opening in inches

T = true stress = strength of material = 300 psi

Substituting in the above formula, transporting, adding and subtracting similar terms we obtain $PV + 24P - 1500V = -12{,}000$, which is the equation of a hyperbola and shows the relation of pressure (causing failure) to the size of the opening.

Fig. 14 shows the experimental curve obtained by the authors in their Fig. 12 and the curve derived by the above equation.

It should be noted in the authors' experimental curve that as the size of the opening approaches 0 the stress becomes infinity, which is inconsistent, while the true stress formula shows that when the opening approaches 0 an external pressure of 500 psi will cause a true stress of 300 psi which will rupture the block.

Also it should be noted that if a diagram of stress was drawn against the size of the opening according to the authors it will follow the pressure curve, being twice its numerical value and varying from —364 psi to —1034 psi to infinity, which is out of the question, whereas in the true stress formula the stress induced to the material is always 300 psi, which is equal to the compressive strength of the material, and its graphic representation will be a straight line parallel to the X axis.

In view of the above facts it appears that the stress analysis of the authors must be in error and therefore their explanations for the so-called anomalous results they obtain seem unnecessary.

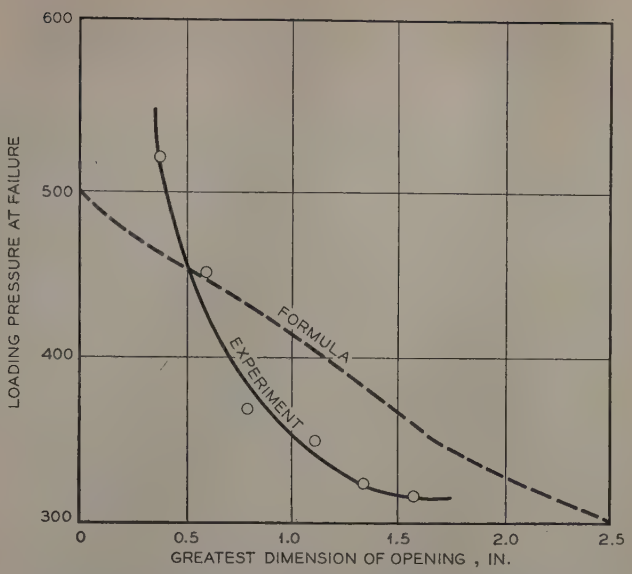


Fig. 14—Comparison of experimental curve obtained by York and Reed in Fig. 12 with curve derived from equation submitted by Zannaras.

It is obvious that the formula applied for the square block also applies for the cylindrical block.

John J. Reed *(author's reply)*—With regard to the above discussion, the *true stress* calculated by Mr. Zannaras is actually an average stress over the entire area of the block at the level of the opening. This takes no account of the known tendency for stress concentration about an opening in a solid subject to external stresses. I refer Mr. Zannaras to the U.S. Bureau of Mines Report of Investigations 4192, *Stress Analysis Applied to Underground Mining Problems,* for an excellent treatment of this subject. Also, we referred in our paper to *theoretically indicated* stresses at the boundaries of the openings, and thus far no attempt has been made to measure the actual stresses in the models. During the coming year I shall be continuing this study, and I plan to measure actual stresses experimentally and compare them with theoretical results. I hope to prepare a paper giving the findings of these studies when they are completed.

B — Minerals Beneficiation

Flotation and the Gibbs Adsorption Equation

by P. L. de Bruyn, J. Th. G. Overbeek, and R. Schuhmann, Jr.

DISCUSSION

W. E. Ewers *(Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia)*—Any attempt to elucidate further the meaning of the contact angle, particularly if it deals with the magnitudes of those mysterious quantities γ_{SA} and γ_{SL}, is certain to receive attention from those interested in the fundamentals of flotation. It is imperative, therefore, that attention should be drawn to a serious defect in the treatment by de Bruyn, Overbeek, and Schuhmann.

Their Eq. 5

$$d\gamma = -\Gamma_x d\mu_x - \Gamma_{H_2O} d\mu_{H_2O}$$

states correctly the relationship between the change in surface free energy $d\gamma$, the surface excesses Γ_x and Γ_{H_2O} of the components x and H_2O and the changes $d\mu_x$ and $d\mu_{H_2O}$ in the chemical potentials of these components. This equation applies for constant pressure and constant temperature and for the interface of the solid *in contact with the solution of X*. The relation remains valid when Γ'_x is defined so that Γ_{H_2O} becomes zero and the equation becomes

$$d\gamma = -\Gamma'_x \, d\mu_x \qquad [17]$$

From this equation de Bruyn et al. correctly concluded that increased adsorption of the collector at the liquid solid interface would reduce the interfacial energy γ_{SL}. Their second conclusion, however, that an increase in the contact angle, corresponding to a greater decrease in γ_{SA} than in γ_{SL}, can be attributed to greater adsorption of X at the solid-air interface than at the solid-liquid interface is quite unjustified.

The portion of the solid surface from which the collector solution has been displaced by air must be considered in terms of the equilibria between the gaseous phase and the surface. If the pressure and temperature

are constant, and if we assume that the collector is volatile, an equation identical to Eq. 5 applies

$$d\gamma_{SA} = -\Sigma\, \Gamma_i^A d\mu_i$$

which allowing similar assumptions to those made by de Bruyn et al. reduces to

$$d\gamma_{SA} = -\Gamma_x^A d\mu_x - \Gamma_{H_2O}^A d\mu_{H_2O} - \Gamma_y^A d\mu_y \qquad [18]$$

Here y indicates the new component, air, in the system and the superscripts, A, on the terms for the surface excess of collector and water indicate that these quantities are not necessarily identical with the corresponding terms in Eq. 5. We can be sure, indeed, that $\Gamma_{H_2O}^A$ is different, because water will be positively adsorbed and $\Gamma_{H_2O}^A$ cannot be assumed to be zero in the presence of air as the *solvent* component. It should be recognized that the chemical potentials here would normally be calculated from the partial pressures of the components but our assumption of equilibrium ensures that these potentials will be the same as in the other parts of the system and are determined therefore by the composition of the collector solution.

It will be noted, however, that in spite of this fact that changes in μ_i in the solution can be identified with those in the gaseous phase, the introduction of the terms $\Gamma_{H_2O}^A$ and $\Gamma_y^A d\mu_y$ makes it impossible to compare Γ_x' and Γ_x^A. Thus the argument of de Bruyn et al. is deprived of its essential step. Their conclusion is therefore not proved, although one must admit the possibility that more collector may be in fact adsorbed per unit area at the solid-air interface than at the solid-liquid interface.

The main objection to their treatment of the system in which the collector is volatile applies equally well to the consideration of an involatile collector.

Essentially the theory of de Bruyn et al. fails because it does not account for the adsorption of water on the hydrophobic surface, and does, in fact, assume it to be zero.

Harkins et al.[21] have measured the free energy change (π_e) when dry graphite is equilibrated with saturated water vapor, and also the free energy change for complete emersion of the graphite $f_{E(SL)}$. These quantities for three samples of graphite each designated by its ash content are set out in Table I.

Table I. Free Energy of Adsorption of Water in Vapor π_e, and Free Energy of Emersion $f_{E(SL)}$, ergs cm^{-2}

Graphite, Ash Content, Pct	π_e	$f_{E(SL)}$
10	56	62
0.46	19	25
Less than 0.004	19	25

The equilibrium contact angle of the pure graphite measured under conditions designed to ensure that the surface had attained equilibrium with respect to the adsorption of water was 85.7°.

Here we see a considerable change in surface free energy (or surface tension) brought about by the adsorption of water vapor on a hydrophobic surface in a system uncomplicated by the adsorption of a collector. It will be evident that changes arising from the same cause must occur also on surfaces where collectors have been adsorbed to make them hydrophobic, and that it is certainly not permissible to ignore these changes in discussing the thermodynamics of the system.

[21] W. D. Harkins, G. Jura, and E. H. Loeser: Adsorbed Films of Water and Normal Heptane on the Surface of Graphite. *Journal of the American Chemical Society* (1946) 68, p. 554.

Masayoshi Wada *(Research Institute of Mineral Dressing and Metallurgy, Tohoku University, Nagamachi, Sendai, Japan)*—By combining Gibbs' equation with Szyszkovski's equation, two equations, Eqs. 19 and 20, expressing the time variation of surface tension due to the change in concentration caused by the preferential evaporation of one component of a solution were introduced according to the assumption that the rate of diffusion of molecules to the surface from the body of solution is small as compared with that of evaporation. It was verified experimentally that the second equation, in which the rate of diffusion of molecules to the surface from the body of solution is assumed to be small as compared with that of evaporation, held good in the aqueous solutions of cineol.

$$H_0 - H = bH_0 \ln (1 - c_1/(a + c_1) \cdot e^{-Kt}), \qquad [19]$$

$$H_0 - H = bH_0 \ln (c_1/a \cdot e^{-Kt} + 1), \qquad [20]$$

where H is the surface tension of solution, H_0 that of pure water, t the time, c_1 the initial concentration, K the time constant, a the concentration coefficient, and b the surface tension coefficient.

The relation between the surface pressure F and the surface concentration u was expressed as follows:

$$F = -bH_0 \ln (1 - uRT/bH_0), \qquad [21]$$

where R is the gas constant and T the absolute temperature. It was verified that the constant b in Eq. 21 varied but little with frothers, and the differences in the surface activity of frothers were mainly due to the ease of adsorption of the molecules of frothers to the surface from the body of a solution.

Further, the relation between the surface pressure and the area S occupied by a gram molecule was expressed as follows:

$$FS/RT = Fe^F/bH_0 (e^F/bH_0 - 1). \qquad [22]$$

It was shown that the equation was applicable to the molecules displaying gaseous state in the adsorbed layer.

By combining Gibbs' equation with Langmuir's adsorption isotherm and Young's equation, Eq. 23 expressing the wetting isotherm in a solid-liquid-gas system was developed, which held good for the wetting of paraffin-wax in aqueous solutions of frothers.

$$H \cos x - H_0 \cos x_0 = b'RT \ln (c/a' + 1), \qquad [23]$$

where x is the angle of contact of solid against solution, x_0 that of solid against pure water, and a' and b' are constants. As the value of $H \cos x - H_0 \cos x_0$ corresponded to the surface pressure exerted by the adsorbed molecules at the solid-liquid interface, it was alternatively called the surface pressure of wetting, by which the wetting power of a surface-active substance was quantified. As the wetting isotherm, Eq. 23, was entirely similar in form to Szyszkovski's equation, the wetting activity and the orientation of frothers at paraffin-water interface was discussed as in the liquid-gas system.

A method capable of measuring directly the wetting tension of solid in a solid-liquid-gas system was developed and the wetting tension-time Eq. 24 was derived from Langmuir's equation of simple adsorption of gas on the surface of solids.

$$(B - B_0)/(B_1 - B_0) = q_1(1 - e^{-nt}), \qquad [24]$$

where B_0 is the wetting tension of the surface not covered by gas molecules, B_1 that of the surface perfectly covered by gas molecules, and B that corresponding to any fraction of the surface covered by gas molecules in a unit area of the surface, q_1 the fraction of the area covered by the adsorbed molecules at the adsorption equilibrium, and n the time constant. It was shown that the wetting tension of crystal glass at the water-air interface decreased fairly rapidly with time and was expressed fairly well by Eq. 24.

In the above discussion, the studies were limited to the simple cases, in which the molecules of pure frothers adsorbed at the water-air, paraffin-water-air, or glass-water-air interface. However, the studies should be extended to still more complicated cases encountered in usual flotation practices.

Thanks are due to Professor Takanori Mayeda, Kyushu University, for his support and criticism.

[22] Masayoshi Wada: *Journal of Mining Institute of Japan* (1944) 60, p. 109; (1949) 65, p. 253.
[23] Masayoshi Wada: *Journal of Mining Institute of Japan* (1943) 59, p. 681.
[24] Masayoshi Wada: *Journal of Mining Institute of Japan* (1947) 63, p. 71.
[25] Mayaoshi Wada: *Science Reports of the Research Institute of Tohoku University* Ser. A (1949) 1, p. 425.
[26] Masayoshi Wada: *Science Reports of the Research Institute of Tohoku University* Ser. A (1950) 2, p. 102.
[27] Masayoshi Wada: *Science Reports of the Research Institute of Tohoku University* Ser. A (1950) 2, p. 127.

P. L. de Bruyn, J. Th. G. Overbeek, and **R. Schuhmann, Jr.** *(authors' reply)*—Mr. Ewers' discussion is much appreciated, as it focuses attention on the problem of defining and measuring quantities adsorbed at the solid-gas interface. Also, Mr. Ewers calls attention to the experimental fact that H_2O is adsorbed on solid surfaces from the gas phase. This fact we recognize fully and took into account but did not discuss in detail in the paper. However, Mr. Ewers objects to 1—our use of the convention $\Gamma_{H_2O} = 0$ for the solid-gas surface and to 2—our conclusion that for simple collection systems there must be greater adsorption of the collector at the solid-gas interface than at the solid-water interface. We find these objections invalid, as shown below.

Mr. Ewers' Eq. 18 may be accepted as basis for discussion. The term $\Gamma_y^A d\mu_y$ is small and unimportant, and is, in fact, nil for flotation with an inert gas ($\Gamma_y^A = 0$) or vacuum flotation (no $_y$-component present). In our analysis we have assumed that only the chemical potentials, μ_X and μ_{H_2O}, change by addition of the collector X to the aqueous phase. The chemical potential of *air* is assumed to remain constant. This is a good approximation provided the concentrations of the collector in the gas and the liquid phase remain small. Finally, if this term were significant, we would expect contact angles and mineral floatabilities to be sensitive to variations in air pressure, which is contrary to experiment.

Thus, Eq. 18 reduces to

$$d\gamma_{SA} = -\Gamma_X^A \, d\mu_X - \Gamma_{H_2O}^A \, d\mu_{H_2O} \qquad [25]$$

Adopting the convention that $\Gamma_{H_2O} = 0$, to which Mr. Ewers objects, Eq. 25 of course reduces to our Eq. 5, and we are led to the conclusion that Γ_X is greater at the solid-gas interface than at the solid-water interface. Our convention as applied to solid-gas interface will be understood more clearly if we proceed from Eq. 25 in a more general way. The Gibbs-Duhem relation for the solution phase, with all solutes at constant chemical potential except for the collector gives

$$c_X \, d\mu_X + d\mu_{H_2O} = 0 \qquad [26]$$

where c_X is the collector concentration in mols of X per mol H_2O. Solving for $d\mu_{H_2O}$ and substituting in Eq. 25

$$d\gamma_{SA} = -[\Gamma_X^A - c_X \Gamma_{H_2O}^A] \, d\mu_X \qquad [27]$$

If we define Γ_X as $(\Gamma_X^A - c_X\Gamma_{H_2O}^A)$, in which Γ_X^A and $\Gamma_{H_2O}^A$ are experimentally measured surface excesses in the gas-solid system, we obtain

$$d\gamma_{SA} = -\Gamma_X d\mu_X \qquad [28]$$

This is the same as the relation obtained by adopting the convention $\Gamma_{H_2O} = 0$. Thus our adoption of this convention means we are defining collector adsorption Γ_X as the total surface excess Γ_X^A experimentally measured less the amount of collector $c_X \Gamma_{H_2O}^A$ which may be considered to be associated with the surface excess of adsorbed water in the same proportions as the two components are present in solution. For practical flotation systems, c_X is generally less than 10^{-4} and often is of the order of 10^{-5} or 10^{-6}. Thus the numerical difference between Γ_X and Γ_X^A, the two definitions of quantity adsorbed, is of no practical significance.

Even if no assumption is made as to the order of magnitude of c_X, this analysis still proves that an increase in contact angle on collector addition requires greater adsorption at the solid-gas interface than at the solid-water interface. Comparison of Eq. 5 for the solid-water interface and Eq. 27 for the solid-gas interface shows that an increase in θ and a corresponding decrease in $(\gamma_{SA} - \gamma_{SL})$ require that

$$(\Gamma_X^A - c_X \Gamma_{H_2O}^A) \text{ solid-air} > (\Gamma_X) \text{ solid-water}$$

or

$$\Gamma_X^A > \Gamma_X + c_X \Gamma_{H_2O}^A \qquad [29]$$

In other words, when the adsorption of water is taken into account, our case for a greater adsorption at the gas-solid surface is *strengthened rather than weakened.*

The authors also wish to express their appreciation of the interest shown by Professor Wada in this problem and his contribution is acknowledged gratefully. Professor Wada, however, stresses the kinetic aspect of the problem more than the thermodynamic approach which was followed in our paper.

Manganese Upgrading at Three Kids Mine, Nevada

by S. J. McCarroll

DISCUSSION

J. Bruce Clemmer, J. B. Rosenbaum, and **C. H. Schack** *(U.S. Bureau of Mines, Salt Lake City)*—We have watched with considerable interest Three Kids development of Manganese Inc. and have been impressed by the ingenuity with which management and staff have solved their many obstinate handling problems.

Mr. McCarroll has been lenient in describing the difficulties faced by the company in developing a process for beneficiating Three Kids ore. The operation, equivalent in size to a 20,000-ton sulphide ore plant, is treating a unique ore by a flotation process pioneered by Manganese Inc. Perhaps the magnitude of the conditioning and flotation problems peculiar to Three Kids ore can be better appraised when it is pointed out that two-thirds to three-fourths of the ore is floated in the rougher step and that the four stages of cleaning which follow are equivalent to floating the whole plant feed two to three times.

Our experience in both laboratory and pilot plant flotation of Three Kids and other manganese ores leads us to agree with Mr. McCarroll that suitable conditioning is a prime requisite for successful oil-emulsion flotation. The difficulties of proper conditioning were demonstrated in pilot plant processing of Artillery Peak manganese ore at our Boulder City, Nev., experiment station. Over three months of experiments and extensive plant revision were required before satisfactory conditioning was obtained in the 25-ton-per-day plant. Such factors as time, type and intensity of mixing, aeration, surface to volume relationship, and power input are still being studied by the Bureau of Mines in hopes of learning how best to condition manganese pulps for oil-emulsion flotation.

Other factors affecting flotation of manganese with oil emulsion also deserve attention. A few of these are the influence of stability and proportion of ingredients in the collector emulsion, the feasibility of grinding

with fuel oil prior to conditioning with an emulsion of tall oil and a wetting agent, the possibilities of stage conditioning with the collector, and the importance of aeration in roughing and cleaning.

The highly friable and porous nature of the Three Kids ore accentuates crushing, grinding, conditioning, flotation, and handling problems. Further degrading of the fine ore apparently occurs even after fine grinding and may account for a part of the difficulty of conditioning and flotation and the large amounts of reagents required.

R. R. Lloyd, Chief, Electrometallurgical Branch, Metallurgical Division, Region III, U.S. Bureau of Mines, has suggested that the difficulty in controlling the temperature and ring formation in the upper end of the nodulizing kiln may be due to the exothermic oxidation of the reduced manganese in the calcined concentrate. If this is so, a remedy would be to permit the calcine to reoxidize prior to charging into the nodulizing kiln. Such a plant change would be difficult but may offer a solution to the continuing problem of ring formation in operating the nodulizer.

We are aware that cursory sintering tests were made by Manganese Inc. prior to selecting the two-stage calcining and nodulizing procedure for lead removal and agglomeration. In view of the still serious difficulties with calcining and nodulizing, it may be pertinent to re-appraise the sintering work. Although the physical strength of sinter is appreciably less than that of nodules, there is reason to believe the sinter strength is adequate. Our small scale tests show that lead can be almost completely removed by addition of about 2 pct salt to the sinter feed. The possibility of volatilizing some manganese along with the lead is acknowledged. The fact that gases from the sinter machine windboxes can be segregated, if desired, may facilitate separation of lead fume from dust. Although major problems undoubtedly would arise in sintering, we confess to being swayed by the prospect of at least having the problem where you can get at it.

S. J. McCarroll *(author's reply)*—The discussion by Clemmer, Rosenbaum, and Schack points out definite lines of investigation which could result in improvements in our process of manganese recovery.

Experimental work on conditioning and grinding is being carried on in both the laboratory and in the mill.

Since the paper was written improvements in operating techniques have resulted in better kiln operation. The idea of exothermic heat resulting from the oxidization of the manganese monoxide, as advanced by Mr. Rex Lloyd, is undoubtedly true and serious thought has been given to the proposal he makes.

With the knowledge we now have of the problems of reagent removal and nodulizing no new installation would be made without thorough investigation of sintering in preference to nodulizing.

A Physical Explanation of the Empirical Laws of Comminution

by D. R. Walker and M. C. Shaw

DISCUSSION

Dimitri Kececioglu *(Allis-Chalmers Manufacturing Co., Milwaukee)*—The idea of applying metal cutting theory to comminution and vice versa is very impressive. Among others, the demarcation of wheel-grinding data into two regions, namely Kick's and Rittinger's, draws attention here. A study of Figs. 6 to 9 shows the following: In Fig. 6, 1 point out of 8; in Fig. 7, 4 points out of 26; in Fig. 8 only 1 point out of 13; and in Fig. 9 practically none out of 13 points fall out of curve No. 2. Also when grinding particles smaller than 5×10^{-5} in. it is difficult to measure very accurately the grinding forces, chip geometry, and particle size. In view of the above it would be difficult to make a positive and distinct demarcation of the wheel-grinding data into two regions, namely, Kick's and Rittinger's.

The idea of using machine grinding to determine the comminution properties of materials is indeed unique. The following cautions come to mind, however, regarding applicability of machine grinding data to comminution:

1—Grinding gives uniform particle size, whereas comminution gives a variety of particle sizes in the same process. The fact that grinding gives uniform particle size is not necessarily an advantage because in comminution the overall energy under actual comminution is desired and comminution invariably results in the production of a variety of particle sizes.

2—In grinding, a small particle is ground off a comparatively large surface. In comminution small bodies are broken down to smaller bodies. This makes it difficult to establish a concept of reduction ratio in machine grinding.

3—The mechanics of machine grinding involve direct shearing action under very high strain and comparatively high compression at the shear zone, whereas comminution involves direct dynamic compression and surface friction with comparatively little strain and shear. Consequently the mechanics of machine grinding appear to be different from those involved in comminution. Hence one might arrive at erroneous conclusions when comminution characteristics are evaluated from machine grinding results.

It is noticed also that functions other than Eq. 16 which do not involve the concept of direct proportionality of useful energy input to new surface area created (Eq. 2 and particularly Eq. 12) may be derived to fit the data of curve No. 2. One such relationship may be $u = ct^{-n}$, where c is a positive constant and n is a positive constant less than unity. This, of course, is made possible because data of a very small range of particle size are being fitted by the curves of No. 2 and the hereby suggested relationship. A sound comminution theory should be applicable to a relatively large range of particle size.

George B. Clark *(Department of Mining Engineering, University of Illinois)*—While the experimental work of Walker and Shaw in this article is of intrinsic value in revealing certain fundamental aspects of machine grinding, milling, and related processes and in adding to the store of information concerning the comminution of solid materials by this particular process, it appears doubtful that the results of their research can be employed to verify either Kick's or Rittinger's theories. Both these laws were originally developed and proposed on the basis of very simple, idealized hypotheses. The creation of parallel idealized physical laboratory conditions which would be required for either their proof or disproof appear to be well beyond the scope of human ingenuity, at least at present.

One great weakness of the imputed verification is that the spread of the points plotted on the graphs is not sufficient to permit accurate curve fitting. The range of particle sizes included in the belt and wheel grinding is not extensive enough to show any general trends, particularly for the horizontal portion of the curves, said to represent Kick's laws.

That the milling, grinding, and turning data obtained by Walker and Shaw for both metals and minerals happen to comply with certain interpretations of these two theories under certain specialized conditions

of the comminutive process is merely fortuitous. The basic factors which govern the energy consumption are concerned with different physical properties than those recognized by the two theories in question; hence their applicability cannot be supported. There are other objections, as well, to the employment of these data to support the much-debated *laws* of crushing. The obvious incompatibility of these results and the development of Kick's theory is that it was devised on the assumption that all strains which contribute to failure in the comminuted material are within the elastic range. Thus the occurrence of plastic flow in the tests in question, which the authors use as a basic reason for drawing a close parallelism between machine grinding and other comminution processes, is one more specific reason why their data cannot be used to verify this crushing theory.

The statement of Walker and Shaw that the machine grinding process offers a precise means for evaluating the grinding characteristics of various materials is also open to question. Outwater and Shaw[8] show that the chip and surface temperatures in machine grinding of metals are very high, high enough to alter the physical properties of the metal. This factor alone would appear to make the applicability of machine grinding as a grindability test a very limited one, that is, restricted to those grinding processes where temperature and grinding speed conditions exist which are similar to those in the actual grinding test. To attempt to apply them to dissimilar tests would be extrapolating through a maze of unknowns to a region of processes at a great distance. Such extrapolation is not justified by the arguments or data presented.

It is also possible that of the energy absorbed by these grinding processes the amount involved in heat losses is so great that the actual energy used in comminution is small enough in comparison to be negligible. These grinding processes, therefore, may be simply measures of deformation rather than comminution, the factors involved in creating particles of "like technological state" being obscured by the energy losses of friction, deformation and other processes, which are not dominating factors in ordinary crushing and grinding.

Another factor which leaves these apparent verifications of the crushing laws open to criticism is that the comminution in each case was accomplished in one step. Such a process ignores the effect of the relative size of the particle being chipped and the body from which it is being separated. In other words the strain both in magnitude and distribution depends upon the geometrical configuration of the bodies being separated as well as the manner in which the forces are applied. One-step crushing or grinding of ores or other solids is an impossibility, except perhaps in the case of explosive action under special conditions. In this case the material might be strained and broken in a single-step process, but even so, the effects of plastic flow behavior of solids under high pressures would come into play. The laws of probability would enter in with respect to particle size distribution, distribution of imperfections in the material, variation of strain distribution from the theoretical, and so forth.

Bond[9] has shown that the mathematical expression employed by Walker and Shaw for Rittinger's theory is in error and that the corresponding curve should be concave downward on a log-log plot. It is asymptotic to a line with a slope of —1 as the product size becomes small relative to the feed size.

For all *stepwise* comminution processes the graphical representation of Kick's theory is also a curve which is concave downward. It could not be a horizontal line because the mathematical expressions proposed by this theory represent the energy of comminution as being a function of the reduction ratio as well as the volume. Hence for the results of Walker and Shaw to be analogous to other comminution processes, the points plotted as being representative of Kick's theory should be shown as points on a family of curves which are concave downward.

To summarize, therefore:

1—The wheel and belt comminution data cannot be used with accuracy to verify Kick's and Rittinger's laws because the physical state of the material is different from that postulated in the development of the laws.

2—The spread of the data is insufficient to justify curve fitting or application of mathematical expressions of these laws.

3—The mathematical and graphical interpretation of Rittinger's comminution theory is in error, and the graphical representation of Kick's theory as a horizontal line is likewise not in agreement with applied interpretations of his hypothesis.

4—The very specialized test conditions involved in belt and wheel grinding would seem to obviate these as standardized grindabiliity tests.

5—None of the laws of comminution have been proved, but they should serve as valuable bases for further theoretical and laboratory investigation.

[8] M. C. Shaw and J. O. Outwater: Surface Temperatures in Grinding. *Trans.* ASME (1952) 74, p. 73.
[9] F. C. Bond: Personal communication.

Fred C. Bond *(Allis-Chalmers Manufacturing Co., Milwaukee)*—The authors have translated considerable study and research in machine grinding operations, principally of metal, into the field of mineral comminution. In doing this they have assumed without any published proof that the depth of cut of a machine tool grinding minerals equals the particle diameter of the ground product, irrespective of the tool speed, grinding surface, and other operating conditions. As would be expected under this assumption, they find large discrepancies between the energy required by a fine grinding wheel and coarser belt-grinding tool.

According to the Rittinger theory the total specific energy varies inversely as the product diameter. Eq. 12 is not a statement of the Rittinger theory because of the term $-u_0$. This term causes the upward curvature of the plotted lines in Figs. 6, 7, 8, and 9. If u represents the total specific energy in reducing from an infinite feed size to product size t the term u_0 should vanish, and the Rittinger plotted line will be straight with a slope of 1:1. If u represents the specific energy increment in reducing from a finite feed size, the term u_0 corresponds to the energy in the feed, and should be added to u, resulting in a curve which is concave downward and which does not fit their experimental data. The error seems to have arisen from their assumption of a positive constant of integration in Eq. 10. The term $V_2—V_1$ in Eq. 8 equals zero from Eq. 7.

When starting with a finite area A_1, volume V_1, and particle size t_1, Eq. 8 should be

$$u = K_2\left(\frac{A_2}{V_2} - \frac{A_1}{V_1}\right)$$

and from Eq. 11

$$u = \frac{K_2}{t_2} - \frac{K_1}{t_1}$$

and

$$u + \frac{K_1}{t_1} = \frac{K_2}{t_2}$$

or

$$u + U_0 = \frac{K_2}{t}.$$

Without evidence regarding the actual particle sizes produced, and with the error in the Rittinger statement, this commentator is unable to accept their conclusions regarding the relationship between the energy input and the three comminution theory criteria of 1—surface area produced, 2—particle volume broken, and 3—crack length formed.

D. R. Walker and **M. C. Shaw** *(authors' reply)*—The authors wish to thank the several discussers for their thought-provoking comments. In answer to Mr.

Kececioglu's observations, we can see no reason why it is more difficult to measure forces and calculate mean undeformed particle shape in fine grinding than in coarse grinding. Although it is true that a range of particle sizes results from comminution, while machine grinding produces particles of essentially constant size, the comminution energy for a range of sizes may be synthesized from the individual values of energy requirements as determined in machine grinding. In comminution there is an appreciable expenditure of deformation energy and friction energy as in machine grinding, so in this sense the two processes are similar.

As stated in the paper, the authors are in agreement with Professor Clark's observation that neither Kick's nor Rittinger's rule has been physically verified, since the original models upon which these rules were formulated are in error. As the title of the paper states, this work is concerned with a physical explanation of the real reasons why the so-called laws of comminution happen to fit comminution experiments. While it is true that extremely high surface temperatures of extremely short duration are encountered in machine grinding, it is equally true that transient high surface temperatures will be experienced in comminution. Professor Clark's statements regarding the difference in physical state because of the plastic deformation of machine-ground particles apparently stems from the common belief that comminution is an essentially brittle process. It has been one of the objectives of this paper to point out that plastic deformation is to be expected in nominally brittle materials when the particle size is small.

Another objective of this paper was to call attention to the physically important variable of particle size rather than surface area. It is thus gratifying to note that Mr. Bond has stated Rittinger's rule in terms of product diameter. Except for a typographical error corrected by Mr. Bond, Eq. 8 is an expression of Rittinger's rule. Mr. Bond is correct in pointing out that the constant u_0 in Eq. 12 should be negative if this equation corresponds exactly to Rittinger's rule. Since u_0 is found to be positive when Eq. 12 is fitted to experimental data, it follows that Eq. 12 does not represent Rittinger's rule, but rather represents a hyperbola which differs by a constant from the hyperbola predicted by Rittinger's rule. This result can only mean that Rittinger's rule fails to agree with experimental data by the amount of the constant. That Rittinger's rule cannot be strictly true can be further observed from Mr. Bond's last equation. If the value of t were made very large, $K_{2/t}$ would approach zero and u would have a negative value. The explanation for this anomaly lies in the fact that Rittinger's rule fails to agree with experimental data by a constant. Since most applications of Rittinger's rule are for energy differences, the offending constant disappears when the difference is taken.

F — Coal

Underground Electrocarbonization of Coal and Related Hydrocarbons

by T. C. Cheasley, J. P. Forrester, and Erich Sarapuu

DISCUSSION

John G. Tripp—The concept of converting fuel deposits *in-situ* to useful forms of energy and chemical raw materials has important possibilities as a means of stretching out our fuel resources. The authors have worked in this field for years and in fact are among the pioneers in developing *in-situ* techniques based on underground electrocarbonization. Their efforts have aroused great interest in this subject and their contributions will undoubtedly gain even more significance as time goes on.

The 4 to 5 billion tons of petroleum are proved reserves, that is, they are completely defined and could be produced economically with present methods of recovery. Reserves of coal, tar sands, and oil shale shown in Table I are not equivalent to *proved reserves* as the term is used in the oil industry. Incidentally, the 4.3 billion tons of oil shown in the tabulation is equal to about 28 billion barrels of oil and, therefore, presumably does not include reserves of natural gas liquids estimated at about 5 billion barrels. The relative comparisons would be more complete if these liquids together with the reserves of natural gas were included in the tabulation.

It is stated in the report that production costs of the order of 10¢ per million Btu's could be expected from a large industrial electrocarbonization installation. The authors state that the operation probably could be operated successfully and at a profit.

I would like to express my view that for the operation to be profitable extremely favorable conditions would have to prevail. For example, the coal seam would have to be thick and close to the surface in order to minimize drilling costs; the deposit should be dry and so situated that there would be little, if any, inflow of ground water; and the seam would have to be relatively gas tight to avoid excessive gas leakage. Notwithstanding the tremendous strides that have already been made, it is my hope that the authors plan to continue their development work. Experiments on underground electrolinking and initial carbonization at Gorgas, Ala., indicate a need for improved control over the operation. Subsequent examination of the underground system at Gorgas has shown that the gasification zone did not always follow a direct path between the electrodes. Undoubtedly the control of the in-situ thermal processes can be improved by further development work.

With respect to the electrocarbonization of oil shale it may be well to point out that the cost of mining oil shale is estimated to be 50¢ to 60¢ per ton. These are low mining costs as compared to coal. Thus it would appear that there is less economic incentive for applying in-situ thermal methods to oil shale than there is for applying these methods to coal. As is the case with in-situ methods for petroleum, the product from the shale having the most economic interest would be liquid oil rather than gas.

The authors point out that for distances up to several hundred miles it is cheaper to transmit energy as electricity than it is to ship energy as coal. This is a very important point, and with coal shipping rates rising periodically, it could prove increasingly important in the future.

I believe that the authors deserve great credit for their outstanding work on underground electrocarbonization. The knowledge resulting from their efforts is of great value to our fuels technology.

James L. Elder—The experimental work conducted by the authors on the electrolinking-carbonization of carbonaceous materials in situ is of great interest. The method is of great importance in the development

of processes for the underground gasification of coal and probably will have application in the treatment of oil shale and oil-bearing sands. However, this discussion is limited to applications of the method towards processes for underground gasification of coal. The procedure is an excellent one for preparing an underground unit for operation. The authors should be congratulated for their experimental development work.

In the paper an effort has been made to develop phrases which adequately describe the phases of the electrical procedure. The term *electrolinking* is used to describe the formation of channels or linkages within the carbonaceous strata in situ. This term is a good one and I believe adequately describes the phenomenon which takes place upon the initial application of an electric current to a carbonaceous deposit. The term *electrocarbonizing* has been used to define the second phase of the process where, following the development of a good electrical path, extensive electrical heating is used to drive off the volatile components of the carbonaceous matter. It also adequately describes the phenomena taking place. A third term, *underground electrocarbonization*, is then used in an inclusive manner to cover electrolinking, electrocarbonizing, electrogasifying, and gasification operations conducted with air, oxygen, or steam without the accompanying use of an electric current. These latter operations cover the true gasification functions whereby a solid fixed carbon is chemically changed to a gas. Application of an electric current alone does not result in gasification of fixed carbon and it can be used only in a secondary manner, to supply heat for the reactions involved. Use of the term *underground electrocarbonization* in an inclusive manner is confusing; it should be applied only where the phenomena involved are completely dependent on action of electric current.

It is stated correctly that it is reasonable to expect gas production costs of the order of 10¢ per million Btu for a commercial plant comprising 30 to 50 coal underground gasification units. This figure is based upon the underground gasification of coal with air following initial electrolinking-carbonization as a means of site preparation. It is expected that in such an operation approximately 1 pct of the net energy produced would occur during the electrolinking-carbonizing phase and 99 pct of the net energy would be from the phase involving gasification with air. In underground gasification of coal the electrolinking-carbonization is by far the best method yet developed for site preparation; it is through use of this method that the most successful gasification operations have been conducted. The quantity production of useful gaseous products, however, appears dependent upon subsequent gasification operations rather than on preliminary site preparation phases.

It has been stated that at the present time there is insufficient experimental data to permit the comparison between uniflow, reversal of flow, or recycling operations. Recycling of product gas during underground gasification might improve the percentage conversion of carbon to carbon monoxide and to date no experimental trials have been conducted that I know of. The improvement would have to be balanced against the costs of recycling which will include repressuring product gas and probably cyclic operations using several unit systems simultaneously. The nearest approach that has been tried experimentally is a cyclic operation involving air blow and steam runs, data for which is given in Table IV. Consideration of the steam run data can be made to estimate the value of recycling product gas, for the operation would have to be carried out in much the same manner. Reversal of flow operations has been tried experimentally on numerous occasions and generally has resulted in a lower quality product. Reversal of flow is usually recommended as a method of decreasing heat losses, but in the experimental trials I believe the opposite results have been achieved.

Continued efforts are being made at Gorgas to determine the location, size, and shape of reaction zones resulting from underground gasification. As stated, tests were made using a modified zero potential electrical method. This has been followed by an extensive drilling program which is still underway. The results of the drilling program do not agree with the electric method. Reference is made to Fig. 3, right; test drilling shows that coal consumption near the input borehole is a maximum and the width decreases as we move downstream towards the outlet. The shape illustrated should show a larger area of coal consumed near the inlet. Using the inlet borehole as a pivot point, the direction of the path which was actually developed is displaced approximately 22° to the right of that shown. The discharge point for the system is probably an old system existing beyond the exhaust borehole indicated and this exhaust borehole is probably not directly connected to the system. The location of the exact discharge point is not yet definitely known. With respect to Fig. 3, left, the reaction path followed has been found rotated exactly 90° to the left, the input borehole being used as a pivot point. In this case the exhaust borehole shown does not appear to be directly connected to the system. The discharge point for the system represented in Fig. 3, left, is actually at the input borehole shown in Fig. 3, right. The test drilling work again shows a maximum coal consumption adjacent to the input borehole.

The changes in path location as well as the existence of carbonized areas influences the resistance-time curves 3, 4, 5 given in Fig. 1. The effective lengths of these paths are no longer the 150 ft shown. For curve No. 3, the length of path must be less than 150 ft and possibly about 120 ft, although it is not now possible to determine this length with any accuracy. For curve No. 4, the length remains 150 ft, for no change in path location has been found. For curve No. 5, the effective length of path must be greater than 150 ft and may approach 175 to 190 ft. It should yet be possible to determine this path length within moderate limits and work is continuing with this objective in mind. It is very likely that the differences in length of path greatly influence the pre-linking time shown in Fig. 1. The history of the electrolinking tests at Gorgas indicates that curve No. 3 of Fig. 1 was probably subjected to more influence by previously carbonized areas than the tests represented by curves 4 and 5.

Longwall Mining and Mechanization with Special Reference To Nova Scotia

by Frank Doxey

DISCUSSION

Louis A. Turnbull *(U.S. Bureau of Mines, Washington, D. C.)*—This paper is a well-organized concise discussion of progress in mechanization at coal mines in Nova Scotia during the last 50 years. It shows the perseverance and resourcefulness of those responsible for the production of high-quality coal essential to steel production in the area. The development of the Dosco miner and its use in longwall mining at the Sydney

coal mines is of particular interest. Some of the operating data given deserve attention. Average productivity of machines and workers is not discussed in detail except that the statement is made that maximum production per shift with the Dosco miner has been 587 tons and maximum weekly production 2860 tons (5 days), an average of 572 tons per shift. If the crew of 55 men on a mechanized unit mentioned near the end of the report is compared with these production figures, the production for the best operating period would be 10.4 tons per man per shift at the face including packing, roof control, and moving conveyors. The statement also is made that there are 96 men in the hand loading crews and 55 men in the mechanical loading crews. Mining with the Dosco miner has reduced the number of men required 43 pct, and if the production for hand loading units vs mechanical loading units is about the same the reduction in labor charges has been substantial.

It is difficult to compare mining operations in Nova Scotia with those in the United States because physical conditions are not at all similar. However, several operators in the United States have experimented with modified longwall systems recently, using European type of equipment. The Meco-Moore machine was used experimentally in West Virginia, the Samson stripper in central Pennsylvania, and the German coal planer is in operation at two mines in West Virginia and one in central Pennsylvania. Data on the operation of the planer in the Pocahontas No. 4 bed in West Virginia are available.[1] A panel 328 x 1455 ft was mined with this device. The coal bed averages 34 in. thick and is flatlying. Maximum production for one shift was 842 tons and average for the entire panel 582 tons per shift for 93 shifts. Average production per manshift was 18.0 tons during planer operation. Including the time spent in developing the panel overall production was 13.0 tons per man per shift which was about 20 pct higher than the average manshift production obtained in conventional mining at this mine. Yielding metal props with fitted steel cap members and wood cribs were used to support the roof. The props were moved forward as the face advanced and a straight break line was almost always maintained. Roof action was good because the roof rock broke and fell almost as soon as the jacks farthest from the face were removed and replaced along the conveyor line. A German-made double-chain panzer conveyor used at the face was at least partly responsible for the good operating results obtained. This conveyor was moved automatically toward the face by pneumatic jack.

Mr. Doxey mentions in the final pages of his paper that improvements in face and intermediate transportation of coal away from the Dosco miner are being studied and will be made. To this extent at least some of our problems are similar. Development of high-capacity loading and mining devices has introduced the problem of moving the coal from the machine to the main transportation system to the end that the loading or mining machine may be kept in operation as continuously as possible throughout the shift.

A number of coal mine operators have expressed interest in modified longwall mining as a means of improving the percentage of recovery of coal and at the same time effecting improvements in productivity per man and machine. It appears that the Dosco miner or some similar device might be suitable for use in the United States where physical conditions of the coal bed and surrounding strata are favorable. At any rate this paper and others presented to the joint Canadian-United States meeting on coal mining undoubtedly will benefit both groups participating in these discussions.

[1] Wilber A. Haley, James J. Dowd, and Louis A. Turnbull: Modified Longwall Mining with a German Coal Planer (Plow) in the Pocahontas No. 4 Coal Bed, Helen, West Viriginia. U. S. Bur. Mines. *R. I. 4922* (1952) 13 pp.

Cleaning Various Coals in a Drum-Type Dense-Medium Pilot Plant

by M. R. Geer, W. A. Olds, and H. F. Yancey

DISCUSSION

John Griffen *(Consulting Engineer, Pittsburgh)*— This paper presents much data offering tentative answers to many technical questions that have been raised by the employment of dense-medium processes and, particularly, by use of the drum-type vessel. It is to be hoped that the Bureau of Mines will publish complete detailed data for the 50 or more tests so that it may be studied and analyzed by everyone interested.

Such study will take considerable time and effort. The writer is therefore obliged to confine his discussion to a few points he has noted as being open to question. The data in Table VI which shows the recovery efficiency for the 1 to ½-in. fraction as 100 pct raised a question concerning the accuracy of these calculated figures; washability data on the washed coal and refuse show that such a figure is impossible. When the ash content of the misplaced material in the washed coal is higher than that of the misplaced material in the refuse, recovery efficiency must be less than 100 pct. A study of the data in Tables III, IV, V, and VI suggest that this error probably arose from slight errors in calculating the data in Table V and more probably from the small scale used in plotting the washability curves from the Table V data.

Using the size consist data and the weight and ash percentages of individual gravity fractions of each size as given in Tables III and IV and the reported recovery of 80 pct of washed coal, a revised Table V was calculated, carrying weight and ash percentages to hundredths of a percent instead of rounding off at tenths. This washability data for each size of Bellingham reconstituted feed was then plotted on large scale washability curves.

From this data the new Table XIV was prepared, which agrees very closely with data shown in Table VI except for two important values, theoretical yields and recovery efficiencies. As will be noted the recovery efficiencies in Table XIV are significantly lower than those given in Table VI.

A study was then made of the data showing distribution of the specific gravity fractions to washed coal and refuse. This study indicated that when this distribution was based on the fractions as tested at 0.1-sp gr intervals the number of points obtained were insufficient to establish the distribution curves with reasonable precision, which in turn would affect the accuracy of the values for the specific gravity of separation and the error areas.

The data in Tables III and IV of the paper and curves prepared therefrom make it possible for the specific gravity fractions to be subdivided further. This was done using 0.05-sp gr fractions and the distribution data so obtained is given in Table XIV, with resulting values for the specific gravity of separation and the error areas.

The writer believes that the derived values shown on Table XIV are more accurate than the correspond-

ing values given in Table VI, but the significant point is that from the same original data derived values are obtained that are different. These differences are such as to indicate lower recovery efficiencies and less sharp separations. This points to the fact that the criteria now being used to evaluate washing performance are dependent upon graphical representation of observed data and that unless great care is used in constructing the graphs, significant errors can arise.

The writer believes this is particularly true in evaluating the performance of heavy medium processes with their much more precise separations. To reduce the chances for errors due to graphing, the float and sink fractions should be determined, certainly close to the probable gravity of separation, at intervals less than 0.1 sp gr, certainly not greater than 0.05 and preferably at 0.02 sp gr. It is believed that tests conducted in this manner, coupled with graphing of data on scales large enough to reduce errors to a negligible value, will render possible derivation of the criteria of washing performance with an accuracy that will give these values practical significance.

The writer has no doubt that most of the conclusions drawn by the authors are valid. However, if the derived values of the other tests are subject to variation in value such as shown by those in Tables VI and XIV, some conclusions may be in error or at least not supported by the original data.

The tentative conclusion that viscosity has no significant effect on washing results does not seem to be supported by the evidence submitted in the paper. The tests 43 and 44 summarized in Table XIII and compared for this purpose both show high error areas. The bath medium of test 44 is supposed to be of low viscosity as it was made up of clean medium, but the specific gravity differential in the bath was only 0.01. Such a low specific gravity differential can be obtained only by grinding the magnetite extremely fine, which entails a high viscosity of bath medium, or by agitating the medium strongly, which impairs the sharpness of separation, or by both. No viscosity figure nor explanation for this low differential is given.

Test 41, which was made on the same coal with a gravity differential of 0.05 (see Tables VII and X) gave much better results. It is also unfortunate that Bellingham coal, which had shown surprisingly high degradation sufficient to affect markedly the error areas, was selected for the comparison on the effect of viscosity or contamination of the medium.

It would appear that a conclusion could have been justified only if the comparison were made on a coal which had no significant influence due to degradation, if the viscosities of the two bath mediums had been determined, and if all other operating variables, including drum speed or agitation, were equal.

The writer thinks it is significant that the gravity differential in the bath seems so important, particularly when separating between 1.55 and 1.60 sp gr. It should be pointed out that no tests are reported for the range from 1.41 to 1.50 nor above 1.60 sp gr. It is therefore possible that a definite gravity differential within the bath has a beneficial effect on performance over a wider range of separating gravities than the authors suggest. However, in view of the error area value of 15 for test 41 shown in Tables VII and VI and the error area value of 20.4 shown in Table XIV, the error areas shown in Table X may be open to question. Should further checking of the data of these tests or additional tests confirm the value of a gravity differential in the bath, it would be a confirmation of the views of K. F. Tromp of Holland, inventor of the Tromp heavy medium process, that the best washing results are obtained in a bath medium with a differential density. A differential in density is obtained by keeping the bath medium both fluid and relatively quiescent. A fluid bath must have a low viscosity. A further requirement for best washing results is that the bath be kept free from congestion with intermediate density materials.

Table XIV. Summary of Performance of Pilot Plant on Bellingham Coal

Screen Analysis	Size Fraction, In. 2 to 1	1 to ½	½ to ¼	¼ to 0	Composite 2 to 0
Feed, pct	22.42	39.90	24.70	12.98	100.00
Washed coal, pct	23.30	41.80	24.30	10.60	100.00
Refuse, pct	18.90	32.30	26.30	22.50	100.00
Ash Content					
Feed, pct	20.47	20.09	24.33	33.11	22.91
Washed coal, pct	15.40	14.20	15.83	13.46	14.80
Refuse, pct	45.43	50.61	55.75	70.14	55.37
Yield of washed coal, pct	83.14	83.81	78.70	65.33	80.00
Theoretical yield, pct*	84.00	84.40	79.40	66.00	80.69
Recovery efficiency, pct	98.98	99.30	99.12	98.98	99.14
Distribution Data, Pct Recovered in Washed Coal					
Under 130		100.00	100.00	100.00	100.00
1.30 to 1.35	100.00	100.00	100.00	100.00	100.00
1.35 to 1.40	100.00	100.00	99.90	99.47	99.92
1.40 to 1.45	100.00	100.00	99.87	99.55	99.93
1.45 to 1.50	97.55	98.21	99.55	97.84	98.42
1.50 to 1.55	42.01	47.68	80.04	80.72	57.32
1.55 to 1.60	4.98	25.38	51.92	56.04	31.91
1.60 to 1.65	0.0	0.0	10.89	17.31	5.18
1.65 to 1.70	0.0	0.0	0.0	12.88	1.77
1.70 to 1.75	0.0	0.0	0.0	3.78	0.57
1.75 to 1.85	0.0	0.0	0.0	4.72	0.73
1.80 to 1.85	0.0	0.0	0.0	10.54	1.79
Over 1.85	0.0	0.0	0.0	0.0	0.0
Specific gravity of separation	1.518	1.520	1.577	1.584	1.537
Error area	10.0	18.8	20.3		20.4
Sink in washed coal feed, pct**	1.41	1.67	1.18	1.31	1.52
Float in refuse, feed, pct**	1.53	1.04	2.00	1.56	0.98
Total misplaced material, feed, pct**	2.94	2.71	3.18	2.87	2.50

* Float-and-sink yield at ash content of washed coal.
** At specific gravity of separation.

The writer regrets that more time is not available for study and discussion of this paper. He urges that the authors arrange for publication of the complete data of all the tests so they can be widely studied and evaluated. The authors are to be commended and congratulated on the ground covered by their paper which the writer hopes will stimulate others to present similar studies of the performance of commercial heavy medium plants.

M. R. Geer, W. A. Olds, and **H. F. Yancey** *(authors' reply)*—The figures in Mr. Griffen's Table XIV differ somewhat from those in the original table VI because he carried his calculations out to the second decimal place and interpolated on the yield-specific gravity curves to simulate the use of additional baths in the float-and-sink tests. These techniques raise the question of whether our chain of data can be stronger than its weakest link: the accuracy with which the yield of washed coal is known. All the performance criteria depend on the yield of washed coal, and generally this figure cannot be determined closer than about 1 pct. Carrying out calculations of yield and efficiency to four significant places simply is not justified by the precision of the basic data.

The practice of interpolating on yield-specific gravity curves is hazardous, as the following illustration demonstrates. In one washing test the usual 0.1 intervals of specific gravity were used for the float-and-sink tests, supplemented by baths of 1.55 and 1.65 sp gr; the distribution data were calculated, using these two additional densities. Then, following the system employed by Mr. Griffen, the yield-specific gravity curves for the washed coal and refuse were plotted, using only the data at each 0.1 interval and ignoring the available data at 1.55 and 1.65. Interpolated values for 1.55 and 1.65 sp gr were read from these curves and the distribution data calculated for comparison with those actually determined in testing. The fol-

lowing tabulation shows that the interpolated values were far from accurate. Errors in distribution amount-

Specific Gravity	Percent to Washed Coal	
	Determined	Interpolated
Under 1.30	99.7	99.7
1.30 to 1.40	99.8	99.8
1.40 to 1.50	99.7	99.7
1.50 to 1.55	97.4	91.3
1.55 to 1.60	59.6	68.1
1.60 to 1.65	29.5	38.7
1.65 to 1.70	17.4	10.0
1.70 to 1.80	6.7	6.7
Over 1.80	0.3	0.3

ing to 9 pct occurred through the interpolation, because of improper assumptions regarding the shape of the yield-specific gravity curves. The likelihood that Mr. Griffen's interpolations were similarly inaccurate is demonstrated by the irregularities in distribution data obtained. For example, his distribution data for the ¼-in. to 0 size shows 10.54 pct of the 1.80 to 1.85 fraction reporting to washed coal, in comparison with a recovery of only 4.72 pct of the next lighter fraction. A similar inconsistency occurs in the composite of all sizes. Such irregularities simply are contrary to the known performance characteristics of washing equipment and point unmistakably to errors—in this case false assumptions about the shape of the yield-specific gravity curves on which the interpolations were made. Thus the authors do not concur in Mr. Griffen's belief that the data in his table are more accurate than the original.

Our observations on the influence of the viscosity of medium were offered as being only tentative, and we agree entirely with Mr. Griffen that the Bellingham coal was not an ideal one on which to judge the influence of viscosity; it simply was the only coal on which the data were available. Additional work now under way will provide a great deal more information on the role of medium properties.

Frontiers in Heat Extraction from the Combustion Gases of Coal

by Elmer R. Kaiser

DISCUSSION

G. A. Vissac *(Consulting Engineer, Vancouver, B. C.)* —Some of the data presented in this paper, in connection with cost studies of washed coals, should be clarified and qualified.

Washing a raw coal down to a low ash content may mean higher losses in refuse, but the cost of the operation may be more than compensated by the increased value of the resultant product. However, careful engineering and accurate machinery are required to insure maximum profits. Washing at a low gravity separation demands a very accurate automatic process, chiefly when the small sizes of coal are being handled.

Coal consumers today are buying mostly on a Btu basis, cognizant of the fact that low ash contents result in lower installation costs and in savings in handling and transportation charges. The attached Table X gives the actual balance sheets of the various cleaning operations corresponding to various ash contents for the two coals presented in the paper, using the old technique of a two-products separation, or the new technique, now commonly used in Europe, of a three-way separation made possible by new methods and new equipment. Savings in capital costs, handling, and transportation have been calculated on the basis of a delivered cost f.o.b. plant of $10.00 per ton of coal at 16.5 pct ash (actual 1953 average price reported by Consolidated Edison, N. Y.), and of $3.50 at the mine also for 16.5 pct ash coal. In both cases this corresponds to values of 12¢ and 4.2¢ per 1 pct of combustible and per ton, respectively, on delivered and on at the mine prices.

As an example, the middle western coal at 16.5 pct ash, raw, washed down to 4 pct ash in a two-way process will give 53.75 pct of clean coal at 4 pct ash and 46.25 pct of refuse at 31 pct ash. The value of the losses will be 0.4625x(100—31)x0.042 = $1.34. Add the cost of washing, capital, and operation, which is $0.35. The total cost will then be $1.69.

But with a three-ways separation we will have 53.75 pct of first-grade coal at 4 pct ash, 37.25 pct second-

Table X. Cost Studies of Raw and Washed Coals at Two Mines. Balance Sheet

Clean Coal		Washing Cost per Ton of Clean Coal	Cost per Ton of Raw Coal, Old Technique			Cost per Ton of Raw Coal, New Technique	
Ash, Pct	Recovery, Pct of Raw Coal		Credits	Debits	Profit or Loss	Debits	Profit or Loss
Midwestern Bituminous Coal							
16.5	100	3.50	raw		0		0
11	91	4.20	0.60	0.455	+0.145		
8.5	86	4.42	0.825	0.535	+0.29		
7.5	83	4.58	0.90	0.632	+0.268		
6.5	79.7	4.75	0.96	0.715	+0.245		
6	78.8	4.80	0.99	0.748	+0.242		
5.5	77.05	4.90	1.02	0.801	+0.22		
5	72.25	5.20	1.00	0.95	+0.05		
4.5	65.55	5.70	0.944	1.22		0.60	+0.40
4	53.75	6.90	0.80	1.69	−0.276	0.604	+0.34
3.5	43	8.50	0.67	2.13	−0.89	0.53	+0.27
					−1.46	0.52	+0.15
Eastern Bituminous Coal							
15.5	100	3.55	raw		0		0
11.0	93	4.12	0.504	0.424	+0.08		
8.5	86.4	4.41	0.675	0.578	+0.107		
7.5	79.7	4.75	0.765	0.79		0.423	+0.342
6.5	71.4	5.26	0.77	1.09	−0.025	0.423	+0.347
6	68	5.50	0.775	1.21	−0.32	0.423	+0.352
5.5	61.4	6.06	0.74	1.37	−0.435	0.423	+0.317
5	54.5	6.78	0.69	1.64	−0.63	0.423	+0.267
4.5	44	8.33	0.58	2.10	−0.95	0.423	+0.157
4	33.5	10.81	0.48	2.50	−1.52	0.423	+0.057
3.5	25	14.55	0.36	2.80	−2.02	0.423	−0.063
					−2.44		

grade coal at 21.7 pct ash, and 9.0 pct final refuse at 72.2 pct ash. The value of the losses will be: 0.09 x (100 — 72.2) x 0.042 = 0.105. Add the cost of washing, which is 0.35, and a penalty on the second-grade coal: 0.3725 x (21.7 — 16.5) x 0.042 = 0.075. The total cost will be 0.53.

The cost of the clean coal, on a clean coal basis, as given in the paper, is of no practical value. It was apparently calculated as follows:

$$3.50/0.5375 + 0.35 = 6.90$$

Credits for savings in handling, transportation, and plant cost are, in both cases,

$$0.5375 \times (16.5 - 4) \times 0.12 = \$0.80$$

Accordingly coal washed down to 4 pct ash may cost $6.90 per ton of clean coal, but it will bring a profit of $0.80 — $0.46 = $0.34 per ton of raw coal with a three-ways separation as against a loss of $1.69 — $0.80 = $0.89 per ton of raw coal with a two-ways separation, as against breaking even, if the coal were sold unwashed, if the market would accept it as such.

Rewashing for a three-ways separation is only worthwhile and practical if and when the primary refuse is under about 55 pct ash.

Removing surface moisture instead of ashes is still the best-paying job, as a 1 pct loss in weight corresponds to a 1 pct improvement in combustible.

In all cases, if a certain amount of foreign materials or of moisture is deemed desirable, it can always be added at the consumers plant, and this will result in savings in handling and transportation charges.

As a conclusion, and in terms of global economy, modern methods of washing down to a low ash content will increase profits and widen markets. When dealing with steam power plants, two different conditions must be considered: using a low ash coal in the remote locations, and a second-grade higher ash coal in the vicinity of the coal preparation plant. This is now the common practice in Europe.

Exploration of the Oaxaca Coal Fields in Southern Mexico

by Luis Toron and Salvatore Cortes-Obregon

DISCUSSION

John D. Price *(Colorado Fuel and Iron Corp., Pueblo, Colo.)*—The paper on the coal fields of the Oaxaca district as prepared by engineers Toron and Cortes-Obregon of the staff of the Bank of Mexico bears witness to the thorough and careful way in which the men associated with this organization perform their work. There is little to be added to their paper in way of discussion other than to confirm and amplify some of their statements.

Since the only extensive and well-developed field of coking coal lies in the northeastern section of the country adjacent to Sabinas in the state of Coahuila, it follows that blast furnace plants would be located in that same region. Two such plants are now operating at Monterrey and Monclova, using coke produced at the Sabinas district mines. But the nearer of these two plants is 600 miles from Mexico City and even farther from the center of population. Transportation of products from these mills to the market area is therefore expensive, both because of the distance and the difficulty of the terrain over which it must be carried. The development of an integrated steel industry closer to the center of population has therefore long been a goal toward which the Mexican technicians have been striving.

While the presence of coal of some grade has been reported in many of the states, and many ideas have been advanced regarding its possible uses in iron and steel production, deposits of anthracite in Sonora and the various coals of the Oaxaca district as reported on in this paper are the only ones that have been explored in a serious manner. The coking coal from the Mixtepec zone appears to offer promise of producing a coke which could be used in a standard blast furnace. Several problems are indicated, however:

1—The ash in the coal is high as mined, but indications are that it can be washed to an ash content of 15 pct with a recovery of 70 pct of washed coal.

2—Such washing would increase the volatile content from 17.4 pct to about 20 pct, and in a byproduct oven this should give a coke yield of close to 80 pct with an ash content of coke under 20 pct.

3—A free swelling index of 5 appears low for a good coking coal, and below that of the coals from the Sabinas district, which show between 6 and 9. But washing of the coal should result in an improvement in this regard; in the United States coals from Utah with an index even lower than 5 have made a usable coke.

4—A coal with volatile as low as 17.4 in raw coal and 20 in washed coal would come close to being classed as a low-volatile rather than medium-volatile coal, and low-volatile coals are notorious for their high expansion properties. Several plants in the United States are making coke from straight medium-volatile coal of 26 to 28 volatile content, and one at Rosita,, Mexico, from coal of 25 volatile. But no plants to my knowledge are using coal as low as 20 volatile. Since the Rosita coal appears to be a borderline coal from the angle of its expansion properties the coking of one of the straight lower volatile must be approached with caution.

5—There are few coals possessing any degree of coking properties which cannot be used in coke production by careful attention to its preparation and blending. The fact that coals of other types are available in this same region make improvement through blending very possible.

6—There are other workable methods of reducing iron ore other than the conventional coke-blast-furnace method. These will not be discussed here but it is known that their use has been considered. The technicians of not only Mexico but also of the other Latin American countries are keenly aware of their natural resources and their national needs. This paper emphasizes the fact that the Mexican technicians are working on their problem and attempting to speed the day of self-sufficiency for their country.

Salvatore Cortes-Obregon *(author's reply)*—I wish to thank Mr. Price for his kind remarks. The Mixtepec coal as shown in Table II has 30 pct ash and a free swelling index of 5, but when the same coal is washed to 15 pct ash it has a free swelling index of 8 to 9 and the volatiles increased from 17.4 to 20.7 pct.

A satisfactory coke has been produced from blends made in the Mexican laboratory using at least 40 pct of the Mixtepec coking coals with the other Oaxaca non-coking coals. Koppers in Germany report good coke obtained from the Oaxaca coal with a blend of 80 pct Mixtepec coal.

Consideration is being given the possibility of using methods other than the conventional blast furnace for the reduction of iron ore near the Oaxaca area; electric furnaces appear promising. The non-coking coals could be used to produce cheap electric energy and the coking coals to make metallurgical coke.

Alkali Reactivity of Natural Aggregates in Western United States

by William Y. Holland and Roger H. Cook

DISCUSSION

Dexter H. Reynolds *(Chapman and Wood, Mining Engineers and Consulting Geologists, Albuquerque, N. M.)*—A number of questions are raised by conclusions and inferences made in the above-mentioned paper. The more troublesome of these concern use of the various pozzolans to combat the deleterious effects of the alkali-aggregate reaction.

The most alkali-reactive materials listed are opal and rocks containing opaline silica. The pozzolans mentioned specifically for use as amelioratives are opaline shales and cherts. These are stated to retard the expansion caused by the alkali-aggregate reaction. Another well-recognized pozzolan is diatomaceous earth, which consists principally of opaline silica.

A pozzolan presumably owes its effectiveness to its high reactivity with the alkaline liquid phase of the concrete mix. It appears reasonable to expect that finely divided opaline silica added as a pozzolan would be more susceptible to reaction with the alkalies present than would larger particles of the same material. The authors report that work with high and low alkali cements indicates that in the presence of alkali-reactive materials, deleterious expansion depends upon the alkali content of the cement. The total effect, therefore, should be more or less independent of the amount of reactive aggregate present, and still more independent of its state of subdivision. The deleterious effects should, if anything, be aggravated by the addition of a finely divided, highly reactive pozzolan.

Further, if the alkali-aggregate reaction is of great importance in the long-term soundness of concrete structures, the addition of a pozzolan to a concrete made with aggregate free from known deleterious materials would be a questionable procedure. The benefits reportedly accruing from such use of pozzolans are greater ultimate strength for a given cement content, increased resistance to deterioration by exposure to sulphate solutions and other mineral waters, and greater resistance to damage by wetting and drying and freezing and thawing. In view of the deleterious effects of highly reactive materials are these benefits ephemeral?

The same considerations apply to another alkali-reactive material, chalcedony, which appears to consist of ultrafine-grained quartz, with opal absent in detectable amounts. Quartz flour is notably reactive chemically and physiologically (cf. Ref. 11 of Holland and Cook's paper), a fact borne out by its effectiveness as a pozzolan, which presumably might be expected to offset the deleterious effects of the presence of chalcedony in the aggregate.

A second question of some importance concerns the reportedly highly deleterious reactivity of acidic and intermediate volcanic glasses, such as rhyolite, perlite, and pumice.

Air entrainment is listed as one of the ameliorative measures to combat the deleterious effects of the alkali-aggregate reaction. The *alkalic-silica* gel formed by the reaction may expand into air bubbles and thus not cause appreciable expansion of the concrete mass. It would appear then that pumice and perlite, particularly perlites of the pumiceous types and other types after expansion, would also tend to counteract the expansion, since these materials consist largely of voids and air bubbles. Certainly this would be expected of structural concrete in which pumice or perlite is used as total aggregate.

Finely ground pumice, perlite, and volcanic ash have been demonstrated to be active pozzolans (cf. *Pumice as Aggregate for Lightweight Structural Concrete* by Wagner, Gay, and Reynolds, Univ. of New Mexico Publications in Engineering No. 5, Albuquerque, 1950). In fact, the term *pozzolan* was first associated with finely divided pumice or volcanic ash. Such materials were used with hydrated lime as the sole cementitious agent in constructing public buildings, roads, and aqueducts by the ancient Romans. The deleterious alkali reactivity of the volcanic glass, itself containing several percent of the alkalies, apparently did not contribute to the remarkable state of preservation of those ancient structures, as exemplified by the Appian Way and the Pantheon Dome.

Still a third question involves the reactivity of constituents of concrete when exposed to various salt solutions. Resistance to deleterious expansion and cracking as a result of contact with mineral waters and its relationship to the mineral content of the aggregate are not mentioned by the authors. Yet the phenomena pictured in Fig. 1, and especially in Fig. 2, appear very much like those caused by exposure to mineral waters. The deterioration of concretes exposed to sulphate waters is generally considered related to the chemical constituency of the cement itself, particularly to the relative amount of tricalcium aluminate contained. Could not many of the ill effects presently blamed on alkali-aggregate reaction really have been caused by contact with sulphate or other salt-containing mineral waters? Or perhaps their use as mixing waters? May not the deleterious expansion be as much a function of the chemical makeup of the cement as it is of the mineral constituency of the aggregate? Would it not be just as important to use alkali-free mixing water as it is to use a low-alkali cement?

It appears obvious that resistance of cements and concretes to sulphate and other salt solutions cannot be left out of account in discussion of deterioration of concrete structures with time. This factor may be of equal or even greater importance than the alkali-aggregate reaction, particularly for concrete subjected to wetting and drying cycles, such as airstrip paving, water-retaining dams, and highway structures.

Another very important factor is called to attention on page 1022 of the article in *Mining Engineering,* October 1953, in that failure of concrete structures may result from poor construction practices and use of too high water-cement ratios. Both of these can contribute remarkably to decreased resistance to attack by sulphate waters, and presumably could have an equally remarkable effect upon extent of damage resulting from the alkali-aggregate reaction.

From the above remarks it appears that while alkali-aggregate reaction may be an important factor in decreasing the useful life of a concrete structure, it is not the only factor involved, and it may not be even a controlling factor. Likewise, many of the phenomena apparently associated with the alkali-aggregate reaction may have resulted from conditions which had little relationship to the alkali-reactivity of a constituent of the aggregate. Certainly if alkali-aggregate reactivity is a major factor in bringing about early failure, one cannot help feeling anxiety concerning the future of the many concrete structures in this country and abroad in which pumice and perlite were used as total or partial aggregates. This anxiety can only be dispelled by calling to mind that among the best-preserved relics coming down to us from ancient times are structures made with mortars containing highly alkali-reactive aggregates.

W. L. Merritt *(Western-Knapp Engineering Co.)*—My interest in the chemical reactions of the alkali in cements began in 1923 when I was superintendent of a diatomite plant at Terrebonne, Ore. Mr. O. Laurgaard, then city engineer of Portland, Ore., conceived the idea of adding pulverized diatomaceous earth to the concrete in a wall the city was building along the Willamette River. At that time the river was highly polluted with industrial wastes and sewage, and existing concrete structures were spauling badly. Mr. Laurgaard firmly believed this deterioration was caused by reaction between the excess alkali in the cement and the river solutions. He believed that the opal content in the diatomite would combine, if added in the mixer, with the available alkali in the cement, thus using up the alkali and preventing the reaction later, and so preventing spauling. This wall was so built; it is still in good condition and many carloads of diatomite for use as a concrete admix were sold. I supervised the plant test work from which we determined that 3 lb of diatomite per 100 lb of cement were required, which I believe, is still about the amount used when diatomite is used for a pozzolan.

My present approach to the problem of alkali-aggregate reaction is not neutralizing the effect in the mixer, but rather, removing the offending reactive materials in the aggregate by the use of heavy media separation, which is proving most satisfactory for this purpose. This takes advantage of the difference in specific gravity of the component materials in the aggregate.

In studying the specific gravities of the minerals now known to be very alkali-reactive, and thus detrimental in concrete, we find that many of them are comparatively light, see Table II.

Table II. Specific Gravities of Alkali-Reactive Rocks

Mineral or Rock	Avg Sp. Gr.	Mineral or Rock	Avg Sp. Gr.	*Na* or *K* Yielding Zeolites	Avg Sp. Gr.
Opal	2.10	Pitchstone	2.40	Analcite	2.3
Obsidian	2.31	Chalcedony	2.62	Natrolite	2.2
Ryolite	2.45	Chert	2.60	Chabazite	2.1
Dacite	2.45				

Most of the accepted desirable aggregate rocks have a specific gravity of from 2.6 to 2.7, so theoretically, aggregates treated in a heavy-media plant operating with a media of 2.47 gravity will be beneficiated by the removal of most of the light undesirable material.

The floatability of a fragment of rock is affected by its porosity, degree of soundness, and its shape. Porous, shattered, thin pieces are not desirable in concrete, so here again the aggregate is improved by removal of this type of material through gravity treatment.

Recent investigations have shown that in the sulphate soundness, resistance to freezing and thawing, and Los Angeles rattler tests, results have been improved on aggregates that were processed by heavy media . One test showed an 82 pct removal of chert and a 77 pct removal of soft material.

In the original paper it was brought out that the zeolites in an aggregate would release sodium and potassium in contact with the hydrating action in concrete, so that the alkali action would be increased when reactive materials were also present. Here again, as shown in the table, the offending minerals have a specific gravity of about 2.2 and can be removed from the aggregate by heavy-media treatment.

As Messrs. Holland and Cook have so ably pointed out in their original paper, no two gravel deposits could possibly be alike in composition. The diversity of source material and the complicated mechanics of their formation render each deposit a separate problem, and often a single deposit will vary greatly in composition both vertically and horizontally.

Heavy-media separation is not a cure-all but a process that will improve some aggregate deposits so they will meet rigid specifications that could not be met without such processing. A system of simple tests on the actual aggregate in heavy liquids will give a very close approximation of what can be done with heavy-media separation. Companies specializing in heavy-media separation systems, such as Western Machinery Co. and American Cyanamid Co., have facilities to make such tests and experienced engineers to interpret the results and make recommendations.

The economics of installing a heavy-media separation plant in a given location like any other mining process depends on many factors, including market, specifications, yardage, distance to a clean deposit, freight rates, and water supply. A well-designed plant handling 300 tph needs only one attendant. Power needs for 300 tph would be about 120 hp including that for conveyors into and out of the plant. Media loss can be held under 1 lb per ton or about two or three cents per ton of gravel. In a deposit with a very high percentage of *reactive* float material the cost of digging and disposing of the waste might become excessive.

In conclusion, while nature has provided some very satisfactory aggregate deposits, many others contain materials that are detrimental to concrete and will not meet rigid specifications. Such deposits must either be abandoned or beneficiated by any one of several approaches, such as 1—improved low-alkali cements, 2—several liquid and dry pozzolans, 3—air-entraining agents, 4—heavy-media separation treatment. The use of heavy-media separation on aggregates is a comparatively new approach, but the two or three plants now operating have been very successful in removing the objectionable material and producing a clean acceptable aggregate. An owner of a deposit that will not pass required specifications should investigate the application of heavy-media separation to his problems. An engineer who is responsible for the specifications on an important concrete structure should protect himself and the structure by investigating the aggregate deposit for alkali-reactive material and insisting on less than destructive percentages.

I should like to suggest that aggregates treated by heavy-media separation be given greater consideration in light of recent developments and the interesting possibilities its known capabilities can uncover.

William Y. Holland *(authors' reply)*—Many of the points raised in Mr. Reynolds' discussion pertain to pozzolanic and related reactivity and therefore do not bear directly on our paper. Some of these questions are discussed in the references cited or may be found in other references mentioned in these papers. It appears, however, that Mr. Reynolds has failed to recall that the fine state of subdivision of the genuine pozzolan as used with portland cement is the one property that assures the material will be beneficial. If the same material of which the pozzolan is composed were of a massive nature such as coarse sand grains or gravel, and occurred, let us say, as 15 pct of an otherwise satisfactory aggregate, it would undoubtedly react deleteriously with the alkalies of a high alkali cement. Although other factors are involved here, one of the principal reasons the coarse reactive material reacts deleteriously is that its reaction is slow, but highly concentrated in certain areas, and does not produce maximum expansion until some time months or even years after the concrete has hardened. On the other hand, the reactive material, if added as a fine powder (pozzolan), reacts rapidly with the alkalies and the reacting areas are very small and dispersed throughout the whole concrete mass, thereby producing, if at all, only insignificant pressures.

We agree that the pumiceous types of aggregates dispel at least some of the reaction pressures by absorbing the gel in voids and gas bubbles. However, if the total aggregate were pumice or perlite, as Mr. Reynolds suggests, their amount would be greater than the *pessimum* and there would not be any swelling in any case, as all the alkalies would be taken care of. A mix such as this would probably be a type of ordinary lightweight concrete. With this understanding, we agree about the Roman roads and aqueducts.

In regard to Mr. Reynolds' third question, we know, of course, that many concrete structures have failed or are failing because of attack of sulphate waters. The examples we chose, obviously, have suffered from alkali-aggregate reaction as determined either by us or by others at least equally competent. We do not favor alkali-aggregate reaction; in fact we could do very well without it.

In Mr. Reynolds' last paragraph, he states that alkali-aggregate reaction is not the only factor involved in decreasing the useful life of a concrete structure. We fully agree. There are probably only a very few concretes that we have examined, and we have examined many, where that statement would not apply excepting laboratory specimens and a few others. It is the degree, or the amount of deleterious effects, including design and formulation, caused by any one or a combination of the many destructive processes that limits the life of a concrete structure.

Mr. Merritt's discussion of the concrete wall constructed along the Willamette River, using diatomite with the mix, is very interesting. Of course, alkali-aggregate reaction may not have been involved here and the problem, therefore, does not bear directly on our paper. Mr. Laurgaard's contention that the diatomite takes up the available alkali is in line, however, with present day beliefs. There are other factors to be considered in the use of diatomite in concrete; some of these are increased plasticity, workability, water tightness, resistance to freezing and thawing, and unfortunately, difficulty with drying shrinkage.

Because of the complex mineralogical nature of most concrete aggregates, we have not tried the heavy media method of separation for these materials. It has been found that most of the deleteriously reacting materials are inherently a part of the individual pebbles of the aggregate and therefore are not amenable to this type of separation even though there may be a slight difference in the specific gravities. If an aggregate occurred in which the deleteriously reacting materials were in individual grains of different specific gravity, this type of separation might be beneficial.

Characteristics of Titaniferous Concentrates

by L. E. Lynd, H. Sigurdson, C. H. North, and W. Anderson

DISCUSSION

D. R. Grantham *(Institute of Mining and Metallurgy, London, England)*—This paper is a very valuable contribution to our knowledge of ilmenite and its alteration products. Two aspects are not treated in the paper and further information would be valuable.

How far are the alteration processes described influenced by the original composition of the ilmenite? The word *gangue* is used but it is not clear whether this refers to other chemical constituents in the ilmenite, e.g., magnesia, or to other minerals loose or in composite grains.

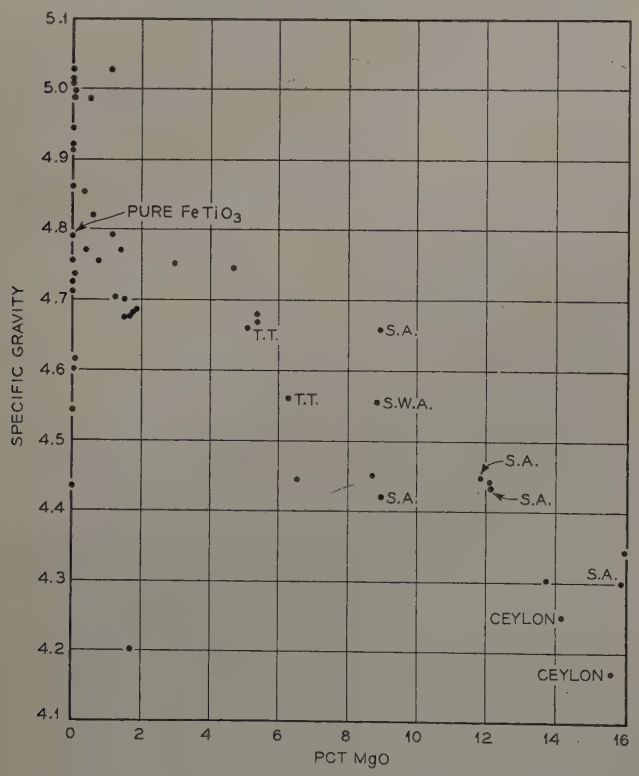


Fig. 17—Specific gravity of ilmenites. (After Grantham.)

Has the work described been related to specific gravities? Pure ilmenite is stated to have a specific gravity of 4.79. Some varieties range up to over 5.0, while high magnesic varieties have a specific gravity ranging to below 4.2. Alteration to hematite might lower the specific gravity and a porous texture due to the removal of iron and the formation of rutile might also lower it.

As specific gravity is a quick method which often gives an approximate indication of composition, data on the above would be valuable.

L. E. Lynd *(authors' reply)*—Because of the way in which beach sand deposits were formed, it is impossible to tell exactly what the original composition of the parent ilmenites might have been. Information on the influence of original composition on the alteration processes must be derived from other sources, perhaps by comparing the alteration products formed from ilmenite in place in a variety of rock deposits, where parent ilmenite and its alteration product can be recognized. No information of this type has yet been brought to our attention.

The material referred to as *gangue* includes all constituents other than oxides of titanium and iron. Nearly all of this was present as separate grains of garnet, augite, hornblende, quartz, feldspar, spinel, or other gangue minerals. Inclusions of gangue minerals within ore grains were rare, and we believe there is little MgO (1 pct or less) or other impurity actually held in the ilmenite structure in any of the concentrates studied.

No specific gravity measurements were made on the samples studied. However, the suggestion that alteration involving loss of iron would lower the specific gravity is a very reasonable one, and is in accord with the findings of Creitz and McVay (Ref. 9 of our paper) who reported two magnetic opaque fractions from Trail Ridge, Fla., to have the following specific gravities and compositions:

Sp Gr	TiO_2, Pct	Fe_2O_3, Pct
3.90 to 4.10	74.2	19.3
4.10 to 4.35	68.8	24.9

The specific gravity of this porous, high TiO_2 alteration product of ilmenite is closer to that of rutile (4.25) than to that of ilmenite. Due to the porosity, it may even be less dense than rutile, although lower in TiO_2 and higher in iron.

Operation of FluoSolids Roaster at Golden Cycle

by Howard R. Keil

Reactor at Carlton mill successfully calcines pyrite-telluride flotation concentrate for cyanidation.

CARLTON mill in the Cripple Creek district, 45 miles southwest of Colorado Springs, Colo., has been in operation for approximately three years, treating the custom sulpho-telluride ores formerly handled by the Golden Cycle mill in Colorado Springs. At the new mill the pyrite-telluride flotation concentrates have been roasted, prior to cyanidation, in a Dorrco FluoSolids reactor, which has proved capable of yielding calcine readily amenable to cyanidation.

Incoming ores are crushed at Carlton to ⅝ in. and sampled in 30 to 75-ton lots before the individual lots are mixed in the fine ore bin. The composite ore then goes to the grinding circuit. Classifier overflow from the grinding circuit is treated by flotation to produce a concentrate containing approximately 85 pct of the values in 5 pct of the weight. The flotation concentrate goes to the FluoSolids reactor prior to cyanidation. Cyanide solution values are recovered by zinc dust precipitation. Flotation tailings are thickened and cyanided, the values from this part of the circuit being recovered from solution by activated carbon.

Gold values in the flotation concentrates consist of a small amount of finely divided metallic gold; the tellurides, sylvanite and calaverite; and gold very intimately associated with the pyrite.

The metallic gold and a part of the telluride values can be extracted from the concentrates by cyaniding without roasting. However, extraction on the tellurides by this method is not satisfactory, and the pyrite values are so intimately associated with the pyrite that very fine grinding and cyanidation for a prolonged period under ideal laboratory conditions does not yield good recovery.

Upon roasting, the tellurides are oxidized to very finely divided metallic gold which is readily soluble in cyanide. The pyrite is oxidized mainly to a porous iron oxide which allows cyanide solution to penetrate to gold particles and take them into solution.

Feeding the Reactor

Flotation concentrates move by gravity to a thickener. Thickener underflow is discharged at approximately 60 pct solids, by means of a diaphragm pump, into a storage and blending agitator. From here the concentrates are pumped to a disc filter. The filter cake, at 84 to 88 pct solids, drops directly into a repulper where it is repulped into a slurry of 78 to 80 pct solids. The repulped filter cake flows into a 6x6-ft slurry agitator from which it is fed directly into the reactor by the Moyno pump.

The Moyno pump has eliminated a large number of difficulties that were first encountered in feeding a wet concentrate to the reactor. On the original installation in Canada a screw feeder was used to introduce the filter cake directly into the reactor. Since the reactor is under a slight positive pressure it was necessary to have a suitable seal to prevent gas escaping from the reactor at this point. When the concentrates produced in flotation were coarse or granular they could be readily fed to the reactor but when fine-textured, clayey concentrates were produced a great deal of trouble was encountered.

H. KEIL, Member AIME, is Mill Superintendent, Carlton Mill, Golden Cycle Corp., Cripple Creek, Colo.

Discussion on this paper, TP 3919B, may be sent (2 copies) to AIME before Jan. 31, 1955. Manuscript, Jan. 14, 1954. New York Meeting, February 1954.

This page of Mining Transactions AIME follows p. 1116. The intervening non-Transactions pages appeared in Mining Engineering.

By repulping the filter cake into a very thick slurry that will just flow, and then feeding this slurry into the reactor with a Moyno pump, it is possible to eliminate these troubles.

The concentrates are fed into the reactor at a point, in plan, directly opposite the main overflow opening and, in elevation, approximately 6 in. above the fluidized bed. A short length of 1½-in. black pipe, flattened and pointing downward at the end at approximately 30° below the horizontal, extends into the reactor 8 in. beyond the inside of the refractory lining. This feed pipe sprays the concentrate down into the fluidized bed. A small amount of compressed air is fed into the feed pipe just before it enters the roaster. This helps to give a spray effect and also serves to cool the feed pipe inside the reactor during shutdown periods.

Roasting the Flotation Concentrates

Temperature control in calcining gold-bearing pyrite for subsequent cyanidation is believed to be of prime importance. In the FluoSolids reactor a very close degree of temperature control is realized with a uniform temperature condition readily maintained throughout the fluidized bed. Under normal operating conditions there is no appreciable temperature difference between different parts of the bed. Heat released during the reaction is immediately distributed throughout the entire bed with no apparent overheating at any point. As long as the sulphur content of the feed is high enough to obtain the desired bed temperature the temperatures can be maintained within a few degrees. Any excess heat released can be controlled by spraying cooling water directly on the fluidized bed or, as is the case at the Carlton mill, by adding additional water into the feed at the discharge of the Moyno pump.

It has been found at the Carlton mill that a concentrate assaying between 19 and 20 pct sulphur must be obtained in order to maintain a self-sustaining roast with a feed containing 20 to 22 pct moisture. However, under nomal operation, a 22 to 23 pct sulphur concentrate is maintained to give a greater factor of safety.

At the present time the reactor roasts between 35 and 40 tpd of concentrates. Since it has a rated capacity of 60 tons per 24 hr, it is operated intermittently 14 to 16 hr per day. It can be shut down and started up without difficulty and without any apparent disturbance in metallurgy or extraction. The bed holds its temperature for a considerable length of time; the reactor has been shut down for periods as long as 36 hr and started again without the addition of fuel. Since there are no moving parts within the reactor itself, all that is required in shutting down is to cut off the feed and air. In starting up, the air is turned into the windbox first and then the feed pump is started.

For longer periods of shutdown an auxiliary fuel oil burner attached to the reactor just above the fluidized bed is used to bring the bed back up to roasting temperature. Also, if sulphur content of the feed falls below a certain point it becomes necessary to put the auxiliary oil burner on for short periods to hold the temperature. At the Carlton mill, however, the reactor has been operated intermittently for periods as long as four months without using any auxiliary fuel.

Since all air is introduced into the reactor by a blower, the degree of oxidation of the calcine can be readily controlled. Calcines ranging from black through shades of chocolate brown to bright red can be produced merely by changing the amounts of feed and air to the reactor. On the black end of the scale practically all the iron is present as magnetite. On the other end of the scale all the iron is present as ferric oxide or hematite. Intermediate products range from red to black in shades of chocolate brown and contain various mixtures of hematite and magnetite.

During the first six months of operation of the reactor a bright red roast was maintained, operating with from 50 to 100 pct excess air. Cyanide residues on the calcines were erratic and were considerably higher than the original tests indicated they should be. The roast was then changed to produce a chocolate-colored calcine, with approximately 1 pct O_2 in the exhaust gases. Since that time very consistent results with good cyanide residues have been obtained. It is believed at the Carlton mill that these results are obtained with the chocolate roast for the following reason. In a single compartment FluoSolids reactor the feed is brought into a zone of maximum temperature almost immediately. If there is a large excess of oxygen present in the freeboard section of the reactor, along with the high temperature, ideal conditions prevail for an instantaneous roast on some of the very finely divided particles. With an instantaneous roast it is conceivable that some of these individual particles reach temperatures several hundred degrees in excess of the bed temperatures and thus lock up some of the gold values to subsequent cyanidation. Holding the excess oxygen to a minimum in the freeboard minimizes the possibilities of instantaneous roasting.

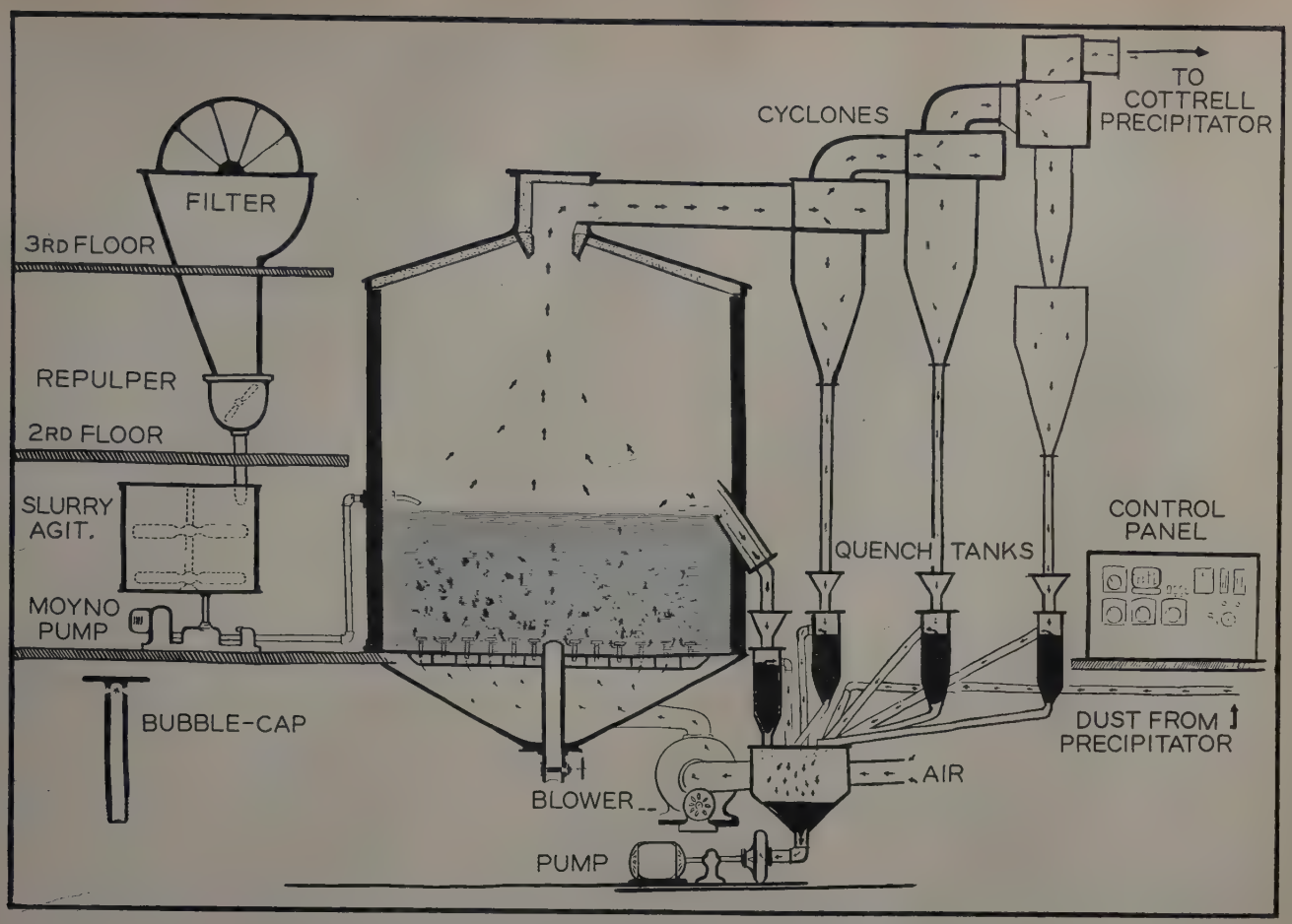
Table I. Operating Conditions and Results of Experiments at Carlton Mill, December 1952

Test number	1	2	3
Color of calcine	Black	Chocolate	Red
Bed temperature, °F	1150	1150	1150
Freeboard temperature, °F	1040	1045	1060
Fluidizing air, cfm	1100	1100	1200*
Feed rate, lb per hr dry solids	4250	4050	3830
Feed, pct solids	79.1	79.5	80.0
Feed, pct sulphur (total)	22.4	22.9	24.3
Feed, pct iron (total)	21.4	22.4	23.1
Feed, pct insoluble	45.6	44.7	41.3
Cooling water, gpm	1.5	1.3	1.9
Stack gas composition			
SO_2, pct		14.9	12.2
SO_3 plus CO_2, pct		1.9	2.1
O_2, pct		0.8	2.1
Excess air (with reference to Fe_3O_4), pct	2.0	4.5	11.6*
Calcine, Fe_3O_4 (calculated), pct	25.35	15.46	1.10
Calcine, Fe_2O_3 (calculated), pct	6.99	15.38	36.03
Calcine, FeS (calculated), pct	1.5	1.5	0.5
Cyanidation test results			
Recovery, Au, pct	88.10	91.40	94.70
CaO consumed, lb per ton dry solids	7.22	6.82	8.61
NaCN consumed, lb per ton dry solids	4.58	3.44	2.58

* Set low to prevent overloading of cyclones.

Along this line, the results of some experiments made by F. L. Bosqui and H. W. Richards of the Dorr Co.[1] working with the staff of the Carlton mill in December 1952 may be of interest. As many performance data as possible were collected under operating conditions adjusted to give black, chocolate, and red calcines respectively. Samples of feed and calcines were returned to Westport for further tests and study. Operating conditions and results are shown in Table I.

It should be noted that although test No. 3 showed the highest gold recovery and lowest cyanide consumption, this test was not a typical red roast. The excess air during this test was only 11.6 pct, whereas normal red roasts are made at 50 pct excess air or better. Sulphating conditions at 50 pct excess air



The FluoSolids Reactor and Auxiliary Equipment

The FluoSolids Reactor or Roaster

1—The cylindrical shell of ¼-in. steel plate, 16 ft 6 in. high, ID 16 ft 6 in. This inside diameter is reduced to an effective diameter of 14 ft, 0 in. in the lower or *bed* section of the reactor by a refractory lining 9 in. thick backed with 6 in. of insulating brick. This heavy refractory lining is carried up from the bottom of the shell for a distance of 5 ft 0 in. or to a level corresponding with the main overflow outlet of the reactor. In the upper or *freeboard* section of the cylindrical shell the diameter is reduced to an effective diameter of 15 ft, 0 in. by a refractory lining 4½ in. thick backed with 4½ in. of insulating brick. Openings are provided through the steel shell and lining for pressure taps, thermocouples, inspection ports, auxiliary burner ports, and feed and discharge connections.

2—The conical shaped windbox attached to the lower end of the cylindrical shell, into which the air is introduced for fluidization. No refractory lining is required in the windbox.

3—The domed top with opening in the center through which the exhaust gases are carried, along with a certain portion of the calcines. The top, like the cylindrical shell, is lined with refractory brick but is backed by a light weight castable insulating refractory cement instead of insulating brick.

4—The constriction plate, fabricated of ⅞ in. mild steel plate reinforced on the bottom side with ½x6-in. steel bars, on edge, spaced on 12-in. centers to resemble a huge waffle iron. The constriction plate has the same diameter as that inside the refractory lining in the lower section of the cylindrical shell, namely, 14 ft, 0 in. There are 134 openings in the plate spaced on 12-in. centers, each opening being tapped with 1-in. standard pipe treads. In each of these openings is screwed a stainless steel *bubble-cap* or tuyere which looks like a piece of 1-in. pipe, approximately 9 in. long, with a 3-in. diam plate, ¼ in. thick, covering the top of the pipe. Immediately under the top plate or cap are four ¼-in. holes, located on quarter-points, through the wall of the pipe. The pipe is threaded on the lower end to fit the threads in the openings in the plate. The constriction plate is covered with two 3-in. layers of refractory brick, laid in such a manner as to leave a 4-in. square opening around each bubble-cap or tuyere. These openings are filled with gannister or crushed fire brick. The bubble caps act as distributing valves for the diffusion of air throughout the bed, and as check valves when the reactor is shut down, to prevent calcine from passing down through the openings in the constriction plate into the windbox below. Through the windbox and constriction plate is fitted an 8-in. pipe with an outside valve for emptying the reactor when necessary.

The Auxiliary Equipment

1—A Spencer turbine type of blower furnishes all the air for the reactor, introducing it through the windbox. The blower is driven by a direct-connected 125-hp, 3450-rpm motor and is capable of delivering 3200 cfm air and 5 psig. The amount of air to the reactor is controlled very readily by a blast gate or butterfly valve on the discharge side of the blower. At the present time 1100 cfm of air are being fed into the wind-box of the reactor at the Carlton mill, consuming approximately 80 hp.

2—A Moyno pump with tool steel rotor and soft rubber stator is used for pumping the raw concentrates into the reactor in the form of a thick slurry of from 78 to 80 pct solids. This pump is driven by a 3-hp direct-connected U.S. Varidrive motor. Rate of feed is controlled by the speed of the pump.

3—Two 42-in. Western Precipitation cyclones and one 24-in. two-tube Western Precipitation multiclone, all connected in series, receive the exhaust gases from the opening in the domed top of the reactor. The connecting ducts, the cyclones, and the multiclone are lined with a castable refractory cement.

4—A Cottrell precipitator receives the exhaust gases from the multiclone before they are discharged up the stack to atmosphere.

5—Four quench tanks receive, respectively, the hot calcine from the main overflow opening of the reactor, the products from each of the two cyclones, and the product from the multiclone. The Cottrell precipitator product is removed from collecting hoppers by means of a screw and is discharged into a stream of water that combines with the discharge from the four quench tanks into a common receiving tank or sump, the contents of which are pumped to the calcine cyanide section of the plant.

would cause a sharp increase in cyanide consumption and a decrease in gold recovery.

There is no direct visual means of observing what is happening within the reactor, but by observing temperature recorders and pressure differential instruments on a control board, one can understand what is occurring and make the necessary adjustments and changes.

Temperature control is simple, consisting of injecting water into the fluid bed or adding additional water with the feed. This is accomplished with an automatic bed temperature recorder controller that automatically opens an air-operated water valve when the temperature reaches a certain point. It has been found at the Carlton mill that a smoother temperature curve can be maintained by operating the cooling water valve manually. Thermocouples are installed in the windbox, bed, and freeboard and in the duct at a point just ahead of the Cottrell precipitator. These four temperatures are recorded continuously throughout any period of operation.

A series of draft gages is mounted on the instrument panel to measure pressure and pressure differentials at and between various points of the reactor and the gas ducts. Gages are connected in such a manner as to measure: 1—total pressure in the wind box in psi (static), 2—pressure drop through the constriction plate in inches of water, 3—pressure drop through the reactor bed in inches of water, 4—pressure drop through the cyclones and connecting ducts in inches of water, and 5—actual duct pressure at a point just ahead of the Cottrell precipitator in inches of water. Any changes in readings of the various draft gages indicate to the operator that some change has taken place within the reactor circuit.

The amount of fluidizing air entering the windbox of the reactor is readily controlled by a blast gate or butterfly valve on the discharge side of the blower. The flow of air is measured by means of an orifice plate and is indicated on a transmitter as well as being recorded on an airflow recorder.

A slurry density recorder-controller was also provided for the reactor feed. This instrument has never worked properly and consequently has never been used. The operator controls the density of the slurry in the agitator by eye. At first, weights were taken on a density scale, but it was soon found that the slurry had a definite appearance as far as fluidity was concerned. Between 80 and 82 pct solids the slurry becomes very doughy and can be balled up and stuck on a wall. At 78 to 80 pct solids it becomes quite fluid and can be readily pumped with the Moyno pump.

The color of the calcine is controlled by the operator by eye. The color of the calcine coming from the Multiclone quench tank is always used as the standard since any change is reflected rapidly and positively at this point. The amount of air to the windbox and the bed temperature are set as constants for the operator. Holding these two conditions constant, he then regulates the feed to the reactor to give the desired color of calcine. This automatically takes care of small changes of sulphur content in the feed and also changes of rates in feed due to consistency of the slurry to the Moyno pump, feed head on the pump, and wear on the pump parts. This method of control has proved satisfactory.

Typical operating data as regards to temperatures and pressures are presented in Table II.

It is imperative that ample dust-collecting equipment be provided when a high-grade flotation concentrate is roasted. Under present operating conditions, approximately 60 pct of the calcine is collected in the main overflow quench tank and approximately 40 pct is carried over with the exhaust gases. The exhaust gases pass through two 42-in. Western Precipitation cyclones and one two-tube 24-in. Western Precipitation multiclone, all connected in series, before entering a Cottrell precipitator. The exhaust gases from the Cottrell precipitator enter a stack and are exhausted to atmosphere. Operation of the reactor was started before completion of the Cottrell precipitator, with only the two cyclones and multiclone in the circuit. However, stack losses were found to be excessive and after several months of operation the reactor was shut down until construction of the Cottrell precipitator was completed.

Table II. Typical Operating Temperatures and Pressures

Air to windbox, cfm	1100
Bed temperature	1150*
Windbox temperature	200
Freeboard temperature	980
Duct temperature (gases entering Cottrell)	590
Windbox pressure, lb per sq in.	2.7
Pressure drop through constriction plate, inches H_2O	24
Pressure drop through reactor bed, inches H_2O	47
Pressure drop through cyclones and connecting ducts, inches H_2O	8.9
Duct pressure (entering Cottrell), inches H_2O	0.6

* All temperatures are in °F.

Table III. Size Analysis and Distribution of Feed and Products, Test No. 2, Table I

Particle Size		Feed, Pct Cumulative Plus	Overflow, Pct Cumulative Plus	1st Cycle, Pct Cumulative Plus	2nd Cycle, Pct Cumulative Plus	Multiclone, Pct Cumulative Plus	Cottrell, Pct Cumulative Plus
Mesh	Microns						
35	417	0.1	0.2				
48	295	0.5	0.9				
65	208	3.1	5.2	0.2			
100	147	11.6	21.0	0.7	0.3	0.6	
150	104	24.5	46.3	1.7	0.6	0.8	
200	74	35.6	65.3	2.6	0.8	1.6	
	60	45.5	81.8	2.7	0.9	4.0	
	40	60.0	94.15	10.5	1.0	14.0	
	30	66.8	96.4	22.0	2.0	31.0	
	20	75.1	97.62	42.0	4.0	59.0	
	10	85.0	98.46	66.5	22.0	86.0	
	8	87.2	98.6	72.0	29.0	90.6	22.2
	5	91.0	100.0	81.5	45.0	96.0	45.0
Weight, pct			59.4	30.75	4.06	2.56	3.23

Table IV. Chemical Analyses of Feed and Calcines, Test No. 2, Table I

	Au Oz Per Ton	Total S, Pct	Soluble S, Pct	Sulphide S, Pct	Total Fe, Pct	Ferrous, Pct	Ferric, Pct	Insoluble, Pct
Feed	8.55	22.9		22.9	22.4			44.7
Overflow	4.50	1.0	0.8	0.2	14.5	2.6	11.9	70.0
1st Cyclone	15.67	11.4	0.6	0.8	40.7	11.3	29.4	31.4
2nd Cyclone	18.04	1.4	0.4	11.0	39.0	14.5	24.5	32.3
Multiclone	17.77	1.2	0.5	0.7	32.7	9.3	23.4	40.2
Cottrell	7.47	2.8	2.7	0.1	32.8	2.9	29.9	36.2

Table III shows a size analysis of the feed to the reactor and of the various calcine products from the reactor as well as the percentage distribution of these products. Table IV shows a chemical analysis of the feed to the reactor and of the various calcine products from the reactor. The data in Tables III and IV were obtained from analyses of samples collected in test No. 2, Table I.

During the two and a half years operation of the FluoSolids reactor there has been very little main-

tenance required. There has been no replacement or repair on the refractory lining. This appears to be almost as good as when it was installed.

After six months of operation the bed was drained from the reactor and all the bubble caps or tuyeres removed for checking. There were no signs of corrosion, but a large number of them were sandblasted on the bottom side of the cap. The ¼-in. holes through the wall of the pipe, immediately under the cap, were all tapered from the inside to the outside when the bubble caps were fabricated. This allowed the air to blast up against the bottom side of the cap, carrying coarse calcine with it and producing a definite sandblast effect. The bubble caps were all repaired by welding and all the ¼-in. holes were filled in so that instead of tapering out in all directions, they sloped downwards slightly. Again, after an additional 14 months of operation the bed was pulled down in the center so that 8 or 10 bubble caps could be reached. These were inspected, in place, and all appeared to be in good condition, no additional sandblasting having taken place.

The reactor feed pipe is replaced every five to six weeks. This is a short piece of 1½-in. black iron pipe, about 18 in. long, threaded on one end and flattened and turned down slightly on the other end.

At the present time the Moyno rotors and stators are lasting three to five months. Adding the cooling water to the discharge of the Moyno pump has seemed to prolong the life of the wearing parts considerably. The rotors have been built up by welding and ground down to give a tight fit on a companion stator. These built-up rotors will ordinarily run for a period of about a month. Some have been built up as much as three times before being discarded.

The quench tanks are all constructed of mild steel and it has been necessary to replace certain parts that have eaten out due to corrosion. The 6-in. seal pipe in the main overflow quench tank must be replaced every two or three months. Seal pipes in the other three quench tanks have been replaced once since the start of operation.

Four diaphragms on the draft gages have been replaced and the vibrator unit on each of the two temperature recorders has been replaced since the reactor was put into operation.

The original steel stack lasted approximately 16 months. As is always the case with steel stacks, there is considerable corrosion above the point where the temperature reaches the dewpoint, resulting from the formation of weak sulphuric acid. A light gage stainless steel stack is being contemplated for the future.

Table V. Roasting Costs, Jan. 1, 1953 to June 30, 1953

Item	Cost per Ton of Head Ore, $	Cost per Ton of Concentrates, $
Labor (operating)	0.044	0.932
Power	0.048	1.052
Water	0.007	0.153
Royalties, materials, and supplies (operating)	0.017	0.381
Repair labor	0.031	0.678
Repair materials and supplies	0.035	0.767
Total	0.182	3.963

Roasting costs are broken down to include all costs starting with the filtering of the concentrates and carrying on through until the gases are discharged to atmosphere. These costs include the filter, repulper, slurry agitator, Moyno pump, reactor, cyclones, Cottrell precipitator, stack, quench tanks, Spencer turbine, and 2-in. centrifugal pump that delivers calcines to the cyanide circuit. Table V gives a break-down of these costs, as referred to above, for a period of from Jan. 1 to June 30, 1953.

The products from the four quench tanks, plus the product from the Cottrell, are all combined in a collecting sump. Enough milk of lime is added at this point to give a concentration of 0.25 to 0.35 lb CaO per ton of solution in the first calcine cyanide agitator. The pulp from the collecting sump, at 8 to 12 pct solids, is pumped by means of a 2-in. Wilfley pump to a 30-ft diam thickener. Thickener underflow is discharged by means of a diaphragm pump, at 40 pct solids, to the first of two 14x16-ft agitators. Aero brand cyanide is fed to the first agitator by a screw feeder to maintain a strength of 1.0 to 1.25 lb NaCN per ton of solution in the first agitator.

Overflow from the second agitator flows by gravity to the first of two 30-ft diam countercurrent washing thickeners. Enough barren solution is added to the second thickener to furnish 540 tons of overflow solution per 24 hr from the first thickener. This solution is clarified and precipitated by the Merrill-Crowe zinc dust precipitation process.

Underflow from the second countercurrent thickener is pumped to a 24x16-ft agitator, the overflow of which is filtered and washed on an 8x10-ft Oliver drum filter. The filter cake is repulped in barren solution and flows by gravity to the flotation tailings cyanide circuit.

Table VI shows some data on concentrate assays, calcine assays, extraction, and chemical consumption over a six-months period.

Table VI. Average Assays, Extraction, and Chemical Consumption, Jan. 1, 1953 to June 30, 1953

Flotation concentrate, Au, oz per ton	5.53
Flotation concentrate, pct sulphur	23.9
Calcine heads, Au, oz per ton	6.68
Calcine tails, Au, oz per ton	0.17
Calcines, pct extraction*	97.5
NaCN consumption, lb per ton calcine	5.19
CaO consumption, lb per ton calcine	9.50

* Additional extraction is obtained on calcines in low grade agitators.

In conclusion, the results obtained to date with the FluoSolids reactor have been very satisfying. The entire system operates smoothly and can be shut down and started up with ease. With the close control of temperature and air input obtainable it should have great possibilities in roasting many materials other than gold-bearing concentrates.

Acknowledgments

The writer wishes to thank Max W. Bowen, General Manager, Charles F. Stanley, Metallurgist, Earl C. Phillips, Chief Engineer and Electrician, and other members of the staff of the Golden Cycle Corp., also T. B. Counselman, F. L. Bosqui, and H. W. Richards of the Dorr Co., for their helpful information, encouragement, and many valuable suggestions in the preparation of this paper.

References

[1] F. L. Bosqui and H. W. Richards: Final Report on Performance of the Golden Cycle Corporation's 14-Ft Diameter Gold Concentrate FluoSolids Reactor Located at the Carlton Mill, Cripple Creek, Colorado M.O. 52239-DD1296 A.J.

[2] Owen Matthews: FluoSolids Roasting of Arsenopyrite Concentration at Cochenour Willans. *Canadian Institute of Mining and Metallurgy Bulletin* (1949) **52**, pp. 97-106.

Diamond Drilling Problems at Rhokana

by O. B. Bennett

Fig. 1—General view of experimental drilling in test chamber.

WHEN diamond drilling was introduced in the Rhokana mines in 1939 it was used principally for pillar removal and for completion of the upper portions of shrinkage stopes which were being affected by increasing pressure. This method of drilling long blastholes proved so successful that it was extended gradually to cover stoping, pillar recovery, and hanging cave work. By 1949 virtually all the production of Mindola and Nkana was being obtained by this method. At the present time 87,500 ft are drilled each month by the 80 diamond drills in daily operation.

Responsibility for control and issue of diamond drilling equipment and crowns, as well as tabulation of all performance figures, was taken over by a specially formed Roto drill department, which also investigated the problems encountered with this new method. To assist this department a fully equipped test chamber, Fig. 1, was established underground where performances of various types of machines and equipment could be studied under conditions as nearly uniform as possible.

Since the establishment of this department, which was eventually taken over and incorporated into the study department, considerable experimental work has been done on every aspect of the subject. The problems can be classified broadly under four headings: improvement of drilling equipment, crown design, machines, and stoping layouts.

One of the major problems with drilling equipment has been to eliminate vibration. Owing to flexing of rods in the hole, severe friction is set up on the back end of the core barrel and on any high spots in the rods, inducing harmonic vibration in the string of rods and causing the crown to chatter against the face. This not only causes premature crown failure but also reduces penetration speeds and increases wear on the machines and rods used.

In the early days, when only holes of EX size were drilled, vibration was largely overcome by periodic greasing of rods and core barrel during each run, but with the change-over to the larger BX hole it became obvious that application of grease by hand was inefficient and time-consuming, and attempts were made to perfect a self-lubricating core barrel. A series of these core barrels was made up and tested and a number of the latest type were used under normal operating conditions, but although footages up to 120 ft were drilled without refilling the overall performance was inconsistent, and the idea was shelved in view of the success of the stabilizer rods referred to later in this paper.

At the same time tests were made with barrels 5 ft and later 6 ft long instead of the normal 2 ft. Although a slight improvement was noticed, greasing was still necessary. It was found that rod vibration increased as the core barrel became worn, and in an early test chamber experiment crowns drilled with a worn core barrel averaged 95 ft with a diamond loss of 4.76 carats, whereas the same type of crowns with a new barrel averaged 228 ft with a diamond loss of 3.13 carats.

Until then all BX drilling had been done with *B*-sized rods, but during a test on a string of BX-sized rods it was noticed that vibration was negligible. Because of the larger surface area of metal bearing on the sides of the hole, however, the friction and resistance of rods of this size rendered them impracticable on any but the most powerful of the machines.

The use of stabilizers spaced evenly along the rods was the next logical step, and for this *B* couplings, see Fig. 2, were set with three tungsten carbide inserts 1 in. long placed around the periphery equidistantly and at an angle of 45° with a right hand lead. These were placed immediately behind the core barrel and then at 12-ft intervals, as it was found that vibration still occurred when the stabilizers were more than 15 ft apart. The effect of these stabilizers was immediately noticeable; holes were drilled with a minimum of vibration, penetration speeds were improved, and as it was no longer necessary to grease the rods there was a marked decrease in the overall drilling time for each hole.

While tests were being made with the stabilizer couplings, experiments were taking place with a set of tapered threaded rods, and because there was marked improvement in efficiency it was decided to incorporate the stabilizers and tapered threading in all new rods ordered for Rhokana. The feature of these rods is that only four full turns are required to tighten the coupling as against nine for the present type of *B* rods. Also, as they are self-centering it is virtually impossible to crossthread them. Each rod has a male 5° tapered Acme thread, Fig. 3, on one end and a female at the other, so that separate couplings are unnecessary, and every fifth rod has an

O. B. BENNETT is General Manager of the Rhokana Corp. Ltd., Kitwe, Northern Rhodesia.

Discussion on this paper, TP 3931A, may be sent (2 copies) to AIME before Feb. 28, 1955. Manuscript, March 1, 1954. New York Meeting, February 1954.

enlarged section behind the male thread into which are sunk the stabilizing tungsten carbide segments.

Although the mines are not yet fully equipped with the new type of rods plus stabilizers, results obtained in areas where the rods are in use are very encouraging. The first sets were issued to a miner whose efficiency was below average. The following figures show the results obtained:

Crown life	*B* rods plus grease	71.6 ft per crown
	Tapered thread plus stabilizers	122.5 ft per crown
Footage per machine shift	Old rods	41.0 ft
	New rods	64.2 ft

In a later major crown test, the footage per reject crown with miners equipped with the new rods was 287.1, whereas that with the old type was 161.5 ft.

To determine the most suitable length further test work has been done on core barrels 5 to 8 ft long. It should be emphasized here that the core barrel used at Rhokana is fitted mainly as a stabilizing section behind a noncoring crown. As rod vibration is induced by friction on the rear end of the core barrel, it was thought that if the core barrel were reduced to 5 in., i.e., by a crown adaptor, and stabilizers were used in the string of rods, friction would be reduced to a minimum. This in fact was the case, but it was found that the deflection of holes of any length was too great to warrant their adoption. As expected, the most accurate holes were drilled with the 8-ft core barrels.

Stones: From the earliest days diamonds used in the crowns have been between 10 and 12 to the carat, and the present average is the grade known as C2, or cast-setting material running 11 stones to the carat. Until recently all stones were from the Belgian Congo, but crowns set with more expensive Premier drilling material have now been drilled both experimentally and on normal run-of-mine work. Footages obtained during initial tests with the better stones were much higher than those with Congo stones, and the costs per foot lower, but in more recent major tests the Premier set crowns have not performed as well as was hoped. Further major tests are being run at present to determine which of the two types of stone is the more economical.

Tests carried out on crowns set with small stones, 20 to the carat, gave very encouraging results, due not so much to increased footage per crown drilled as to the decreased diamond losses, which were in proportion to the weight of stones set. Five crowns with stones 40 to the carat set in an extra hard matrix were promising, although four failed prematurely because the center collapsed. The fifth, however, reached a remarkable footage of 506 ft, indicating that further tests along these lines are warranted. The size to be used is governed largely by availability, however, and as stated earlier, stones averaging 11 to the carat are set in the normal mine crowns at present.

The theory that stones become brittle and break during drilling after frequent salvage and re-use was tested in an early experiment using a particular set of stones as many times as possible. A study of results obtained indicated that the structure of some stones is naturally stronger than that of others, and that fatigue is not an important factor in stone failure, as there was a marked decrease in failures after each re-use. Further work is being done on this.

Crown Design: A careful study of each batch of crowns received from the manufacturers revealed considerable variation in the pattern of stones on the crown face.

Until then the pattern of stones on the crown had been left to the die puncher at the factory, and there was no way of knowing beforehand whether crowns from any particular master die would perform satisfactorily. It was obvious, therefore, that some method should be devised whereby the stone loading of a crown face could be determined and measured against a known series of *minimum loadings.* An official on drilling research worked out a means of calculating the number of cutting points per unit area at fixed intervals across a crown face. These figures are plotted against an average minimum loading line determined by a study of the characteristics of a number of different crowns whose loading lines had already been plotted. Using this method it was possible, by working from the master die, to determine whether crowns made from that die would have any weak spots.

It was by no means easy, however, to work back from the minimum loading line and produce on the drawing board a crown theoretically having no weak spots. A second method has therefore been evolved by a member of the manufacturing company by which the whole stone layout is plotted on a drawing and then transferred to a die plate by a photographic process perfected in the Diamond Research Laboratories.

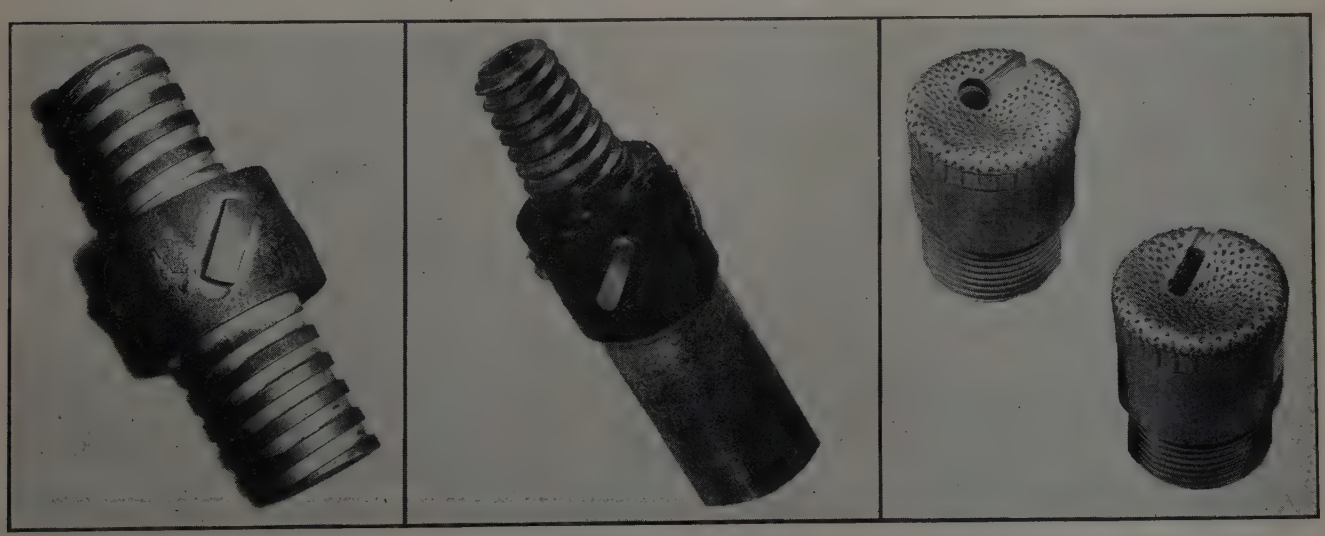


Fig. 2 (left)—The *B* coupling with tungsten carbide insert. Fig. 3 (center)—Tapered threading and stabilizing section on new rods. Fig. 4 (right)—The BX crowns used at Rhokana mines.

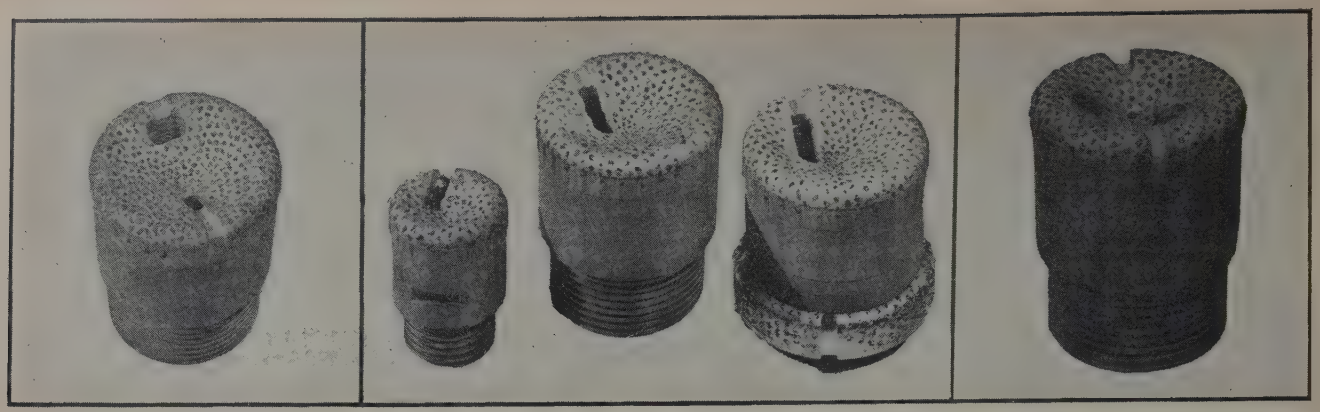


Fig. 5 (left)—Experimental BX crown showing layout of water holes. Fig. 6 (center)—Types of crowns in use at the present time. Fig. 7 (right)—Experimental crown with offset water grooves.

One major test has been run on a series of crowns that had been punched arbitrarily and checked by the first of these methods. A second test is now in progress. This particular series, the BX2 shown in Fig. 4, was chosen because its loading graph most nearly approached the theoretical line, although a weak spot was noticed during the test. This weakness is now being remedied in the BX5 crown by an increased number of stones in the underloaded area. Results were noticeably better than those obtained with the crowns previously used, the footage per reject of the BX2 rising to 163 ft from 119 ft.

BX4 test crowns made to a drawing board design are now being tested. First results indicate that they contain no inherent weaknesses and that a large-scale test is warranted. These crowns, Fig. 5, have two water holes in place of the normal one. In this design the distance between the stone centers is 125 pct of the diameter of a stone, and each stone is slightly further from the center than the one ahead of it. The diamonds thus lie in lines curving out and away from the center.

All recent test work has been done on the BX noncoring crown, but the use of a pencil coring crown offers many attractions, and a number of tests were run at Rhokana to find a suitable type for use at the mines. Advantages are: 1—reduction in the weight of diamonds set, with consequent decrease in stone losses; 2—less rock to be cut, therefore faster speed; and 3—elimination of the vulnerable center portion. Results were disappointing, however, and trouble was caused by breaking and jambing of core in the core barrel.

The only coring crowns now used, those giving a 1⅝-in. core, are drilled in ground too hard for a noncoring crown. In an attempt to perfect a continuous coring operation extra hard matrix was used in these crowns, together with rods flush-jointed on the inside, but no notable success was achieved.

In addition to the BX-size holes being drilled on the mine larger holes are used for hanging-cave work and in some cases for slot blasting. These holes were NX size, but as the largest explosive sticks available were only 2½ in. the latest holes are all 2¾ in. diam. All these are drilled with a standard BX pilot followed by a reamer, but some full-size noncoring crowns with the latest stone pattern (BX4) are now being manufactured for test purposes to determine which method is the more economical, see Fig. 6.

Early experiments proved that to prevent any discoloration due to heating the waterflow over an EX crown should be 4 gpm, and 6 gpm over a BX noncoring crown. However, with the introduction of more powerful machines, it was found that despite a flow in excess of these figures certain portions of the crown face were becoming discolored or damaged by heat. It was clear, therefore, that with the one hole crown, waterflow across the face was not sufficient, and tests were made, first by increasing water pressures and second by using specially made crowns.

These tests showed that there was little to be gained by increased pressure. Optimum results were obtained with water at 130 lb per sq in. Two-hole crowns gave greater and more even flow of water than the single-hole, and heating was not evident even when water pressure was dropped well below average. In a further test with crowns with two holes but with offset water grooves not connected to the water holes, see Fig. 7, sludging was excellent for the first few feet but fell away rapidly as chippings packed in spaces between the stones.

It is not clear from these tests that better sludging is to be obtained with two water holes and grooves in the crown face, and when the present series of tests is concluded it is intended that a major test will be run on the BX4 two-hole design.

The crown profile has been altered considerably over the years, but it has now been standardized with a 25° inner slope. It was found that with a more acutely angled or flatter face performances were poor and holes deviated. A deeper angle presented manufacturing difficulties.

Little work has been done recently on matrix materials. The tin bronze used exclusively in crowns supplied to this corporation apparently satisfies the requirements sought, resistance to abrasion by the sludge and ability to hold the diamonds securely.

As drilling experience has increased, the trend on the mine has been towards drilling larger blastholes to reduce diamond drill footages and development work required and at the same time to increase the operators' productivity. Consequently it has been necessary to find machines of suitable qualities for the heavier work. Eighty diamond drills, as well as 45 percussion machines, are now empolyed to break 350,000 tons monthly, whereas 320,000 tons were broken previously by 34 stopers.

Four main problems in machine design have to be considered: spindle speeds, power, types of feed, and the pressure on the diamond crown most suitable for any particular ground. Little work has been possible locally on the fourth problem.

The original machines used for EX drilling ran between 1600 and 1800 rpm, but slow speed drilling tests in South Africa were so successful that experiments were carried out here. These tests were made

with BX-size holes, and as it was found that rod vibration was noticeably less than at higher speeds and performances better, all new machines are designed to operate between 900 and 1250 rpm. It is of interest to note, however, that at 2500 rpm a standard EX noncoring crown gave a life of 435 ft as against the normal 111 ft and penetration was 60 pct faster, but owing to the excessive machine and equipment wear these tests were abandoned.

With the larger *Q* holes and the longer BX holes now being drilled, it was found that the machines on the mine were underpowered. Twenty-five 18-hp piston machines have now been introduced and are operating satisfactorily. A major advantage of these machines is that they are fully reversible, and with the new rods rod-changing is a matter of seconds.

Increased air consumption with these larger drills has now diverted attention to the possible advantage of drills with a different type of drive, and a certain amount of test work on an electrically driven hydraulic oil machine has been carried out in the test chamber. With the necessary oil tanks and pumps the drill was cumbersome, but results were so encouraging that a second prototype has been made.

This second machine incorporates an hydraulic feed system, and it is expected that the steady pressure on the crown will improve both crown life and drilling speed. All the present machines are fitted with automatic screwfeeds, as the hydraulic feed cylinders are both heavy and vulnerable to abuse.

How Diamond Drilling Influenced Mining at Rhokana

Considerable advances have been made in stoping layouts since diamond drills were first introduced. The original method was to drill EX fanning rings at 5-ft intervals along subdrives mined at 25 ft (later 50 ft) vertical intervals, Fig. 8, but when BX drilling was adopted parallel holes were drilled from crosscuts driven every 10 to 12 ft from the sublevel on the hanging wall side. The sublevel vertical interval was now increased to 70 ft, and the latest method of BX fan drilling from the hanging wall drive has eliminated some 150 ft of crosscut development for each stope. In this method, which is proving the most efficient to date, the BX split rings, Fig. 9, are drilled with 6-ft burdens, but in such a way that the holes in the second ring cover the spaces in the first ring. The toe-burden in each split ring is 24 ft.

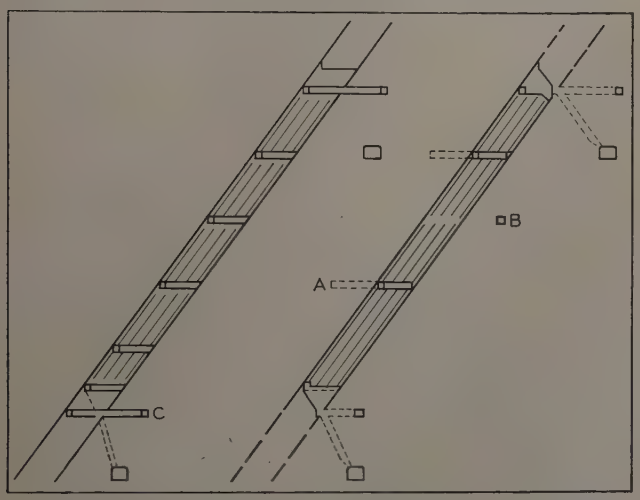


Fig. 8—At left, old method of parallel drilling from crosscuts on sublevels spaced at 50-ft intervals. At right, later method of parallel drilling from crosscuts on two levels only. *A*, crosscut for hanging wall caving; *B*, pillar recovery drive; *C*, grizzly level.

Item	Parallel Ring	Split Fan Ring
* Footage drilled	1050	1087
* Footage charged	1002	795
* Cases explosives	37.11	29.44
Tons produced	6660	7910
Tons per foot drilled	6.34	7.28
Tons per case explosive	179	269

* Excluding any easer holes.

In each method, a slot is mined across the orebody from foot to hanging wall and the rings are blasted successively into this.

In the last 15 years, the use of long blastholes for rock-breaking has revolutionized the system of mining at Rhokana, and the tonnage produced per miner has been more than doubled. At the same time, it has been possible to evolve a mining method that is very much safer than the original shrinkage stoping system, as all work is now carried out from positions well clear of the open stopes.

In any system, however, the overall costs are of major importance, and with the high price of diamonds and the rising world prices of labor and material, the continued employment of diamond drills for long blasthole work at Rhokana has been made possible only by continuous research and test work. With the improved efficiency resulting from the experiments described, it is confidently expected that diamond drilling will play a major part in mining operations at Rhokana for many years to come.

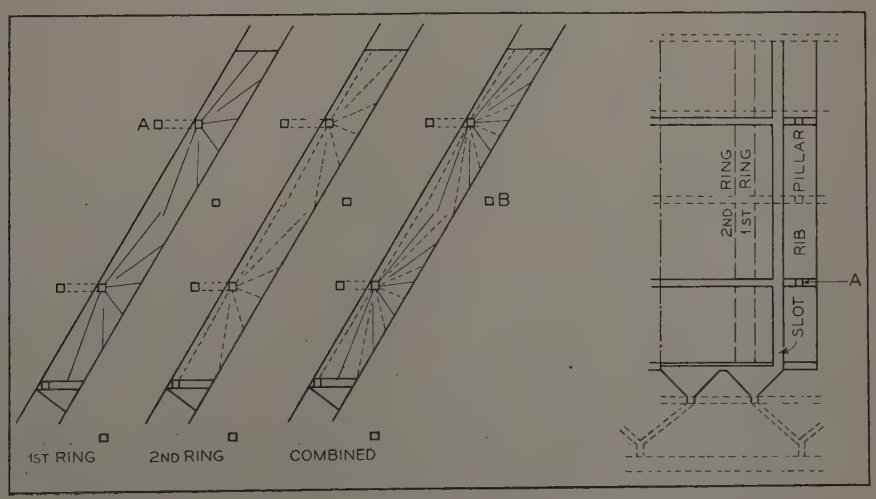


Fig. 9—At left, split ring stoping cross-section shows method of drilling from hanging wall drives. *A* indicates hanging wall crosscut for caving. *B* indicates pillar recovery drive. At right, first and second rings of longitudinal stoping. *A* indicates hanging wall crosscut.

Colorado Fuel & Iron Corp.

Improvements in Plant and Operations At Pueblo Coal Washery

Making maximum possible use of available equipment and material, CF&I placed a high-efficiency, high-capacity washery unit in the existing buildings to gain simplified operation, reduced manpower requirements, raised efficiency, and boosted production.

by J. D. Price and W. M. Bertholf

THE central washing plant of the Colorado Fuel & Iron Corp. was first operated in 1918 to furnish coal for two 60-oven batteries of Koppers design. Prior to that time the coke for the blast furnaces had been secured from a number of beehive operations, some with their own washeries.

The original washery had four Elmore jigs, which did not do as well as had been expected. In 1923 the jigs were taken out and replaced by Deister Plat-O tables, with various changes in auxiliary equipment. In 1936 the throughput of the plant was considerably increased by the installation of vibrating filters for recovery and drying of fine coal, thereby reducing a large recirculating load of fine coal which passed through the screens of the Carpenter driers.

In the late 1940's it became evident that something would have to be done to keep the washery abreast of progress at Pueblo and other washeries. Coal requirements were nearly two and one-half times the original requirements of the 120 small ovens, as additional ovens had been built in 1930, 1938, and 1945, whereas no additional coal washing capacity had been added since the 1936 modifications. Much of the equipment, moreover, had seen better days.

One answer to the problem was to build a new and completely up-to-the-minute plant, with separate sections for coarse, medium fine, fine, and very fine coal. On the other hand, it might be financially more profitable to place a high-efficiency, high-capacity unit in the existing buildings.

The washing problem at Pueblo is not unique, but it is approached differently. There are no established specifications for coke ash and sulphur. The plant is operated on the principle that if it is more economical to wash it out than to slag it out, wash it out; otherwise, send it on to the blast furnaces. With coals at the Pueblo plant costing what they do, the benefits of washing at a low gravity are questionable. Hence the primary problem is to eliminate the high-ash components and retain all the low and medium-ash components. Sulphur is no problem at all, averaging 0.6 pct in the coke.

Since a good proportion of the free impurity in the coals is as large as the coal or bone or larger, it appeared that it would be safe to go to jig washing and discontinue crushing the coal before washing, as had been the practice with the tables. This would boost the throughput. It might also decrease manpower requirements and washing costs considerably.

It was assumed at the start and has been demonstrated in the first year of operation that a jig will do as well on the fines as the tables had been doing. It would not be necessary to discount any gains due to increased efficiency in washing the coarse material because of incapacity to wash the fines.

By washing at $-2+0$ mesh it would be possible to dewater, by screening, a considerable tonnage satisfactorily, for example, at $-2+\frac{3}{8}$ or $-2+\frac{1}{4}$, and then crush it in a hammer mill. The $-\frac{3}{8}+20$ could be dewatered in centrifugal driers and the remain-

J. D. PRICE and W. M. BERTHOLF, Members AIME, are, respectively, Superintendent and Preparation Engineer, By-Product Coke Plant, Colorado Fuel & Iron Corp., Pueblo, Colo.

Discussion on this paper, TP 3922F, may be sent (2 copies) to AIME before Feb. 28, 1955. Manuscript, Feb. 2, 1954. New York Meeting, February 1954.

ing fines handled as before in the old fine coal circuit until it was possible to work out something better, if that should prove necessary. The net result would be that 1—large rock would be rejected immediately and completely; 2—large coal would be dried very economically and then crushed in a machine designed for the job; 3—the medium-size coal would be dried in a few efficient units; and 4—even with increased throughput, the total quantity of fines to be handled would not strain existing facilities. If all went well, there would be a continuing saving as compared with the cost of operating the old plant, in addition to the fact that more coal would be washed than the present ovens could possibly carbonize, and there would be ample time for repairs and maintenance on down shifts.

Installation of the new equipment, originally planned for late fall 1951, was started early in 1952. By late May there were two jigs in place, and enough auxiliary equipment to operate after a fashion without interruption of coke plant operations. Because of the steel strike all operations were suspended for two months, and the contractor was relieved of the responsibility for completing the job. This took the company several months, and it was close to the end of the year before the installation was completed.

The data presented here are intended for comparison of results before and after the installation, with reasonable accuracy and in enough detail to show that expectations will probably be realized in full. Charts show yearly averages from 1930 through 1953 and monthly averages from August 1952 through December 1953. Particular attention is directed to the last six months of 1953 as compared to the previous year of operation of the new plant as well as the old plant record.

Rearrangement of Washery

Fig. 1 illustrates the essential nature of the changes made in the washer building. Twenty-three primary tables as well as four rewash tables located on a lower floor were replaced by two 84-in., 3-compartment, 6-cell air-operated Jeffrey jigs, with a proposed 32 pct increase in hourly throughout. The area above old tables 15, 16, and 17 has been utilized for a modern electrical control room and on lower levels there is a considerable increase in the amount of free space. Six old Carpenter-type driers were replaced by 3 CMI Model E driers, with a saving in floor space which has since been converted to a weld shop for the coke plant.

The old drainage conveyor for washed coal has been removed and the collecting sump for fine coal reduced in size. Some of the trench system has been covered over, greatly facilitating housekeeping. Resulting space will be used to good advantage in an extension or replacement of some of the fine coal recovery equipment. The old boiler coal and refuse pits, at first floor level, were filled and also covered.

The dewatering screens, 5x16-ft Allis-Chalmers Low Heads, now occupy space formerly taken up by tables and launders. Below and west of these units the rewash floor was extended to take the clean coal crushers, Jeffrey Flex-Tooth, 24x42-in. This is the only part of the building which appears to be crowded, but there is ample room for any necessary maintenance work.

The only addition to the building is a small blister on the east side, housing the blowers and feed

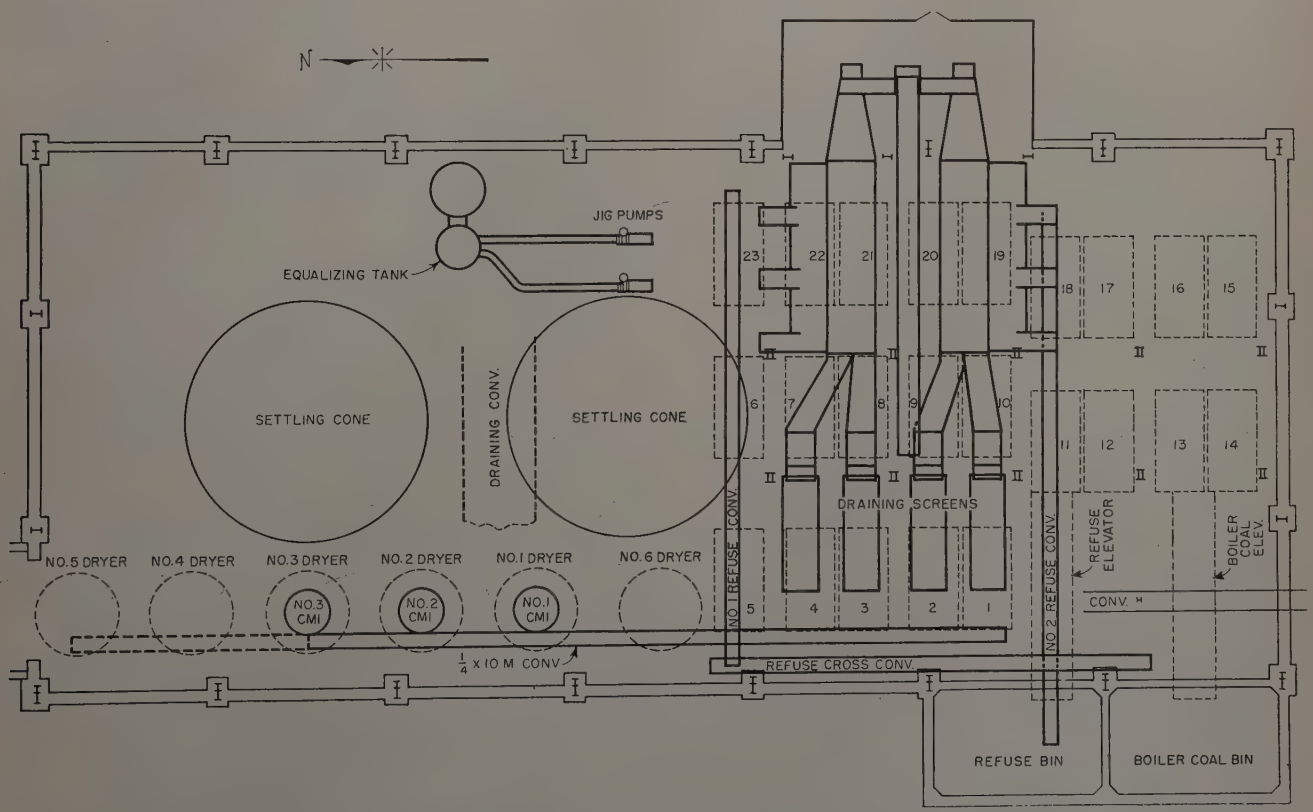


Fig. 1—In the washer building twenty-three primary tables and four rewash tables have been replaced by two 84-in., 3-compartment, 6-cell air-operated Jeffrey jigs. With the jigs coal is no longer crushed before washing as was the practice with the tables, since much of the free impurity in the coals is as large as the coal or bone, or larger. It has been demonstrated in the first year of operation that jigs can do as well on the fines as the tables were doing.

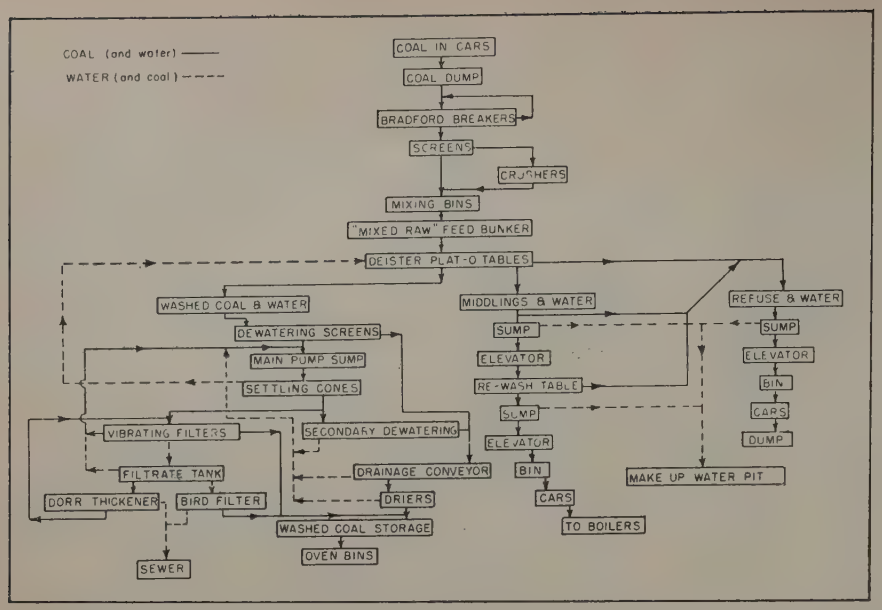


Fig. 2—This flowsheet of former operations should be compared with the new arrangements illustrated in Fig. 3. The Bradford breakers, the screens, and the roll crushers have now been eliminated, and coal is being dumped and sent directly from the receiving hopper to the appropriate bin.

sluices for the jigs. This has 19-ft clearance above the railroad tracks.

Building Schedule

The notations accompanying the contractor's* drawing shown in Fig. 1 were as follows:

"In general, the plant will operate at normal capacity during erection of Part I. While No. 1 jig (Parts II and III) is being erected, 14 tables will operate. After No. 1 jig is in operation the remaining tables will be removed and No. 2 jig will be erected (Part IV).

"Part Ia—Install three CMI driers. Remove one old drier at a time, install new ones, and operate with temporary chutes. With three driers in operation, remove remaining old driers and erect new platform with present distributing conveyor in operation. At a convenient time, remove present distributing conveyor and install ¼-in. by 10-mesh conveyor, removing No. 6 drier and supporting structure first.

"Part Ib—Erect new structure for blowers and jig chutes and sluices. Erect conveyor and conveyor gallery to jig sluices.

"Part IIa—In preparation for erecting No. 1 jig, alter water line to tables.

"Part IIb—Relocate present clean coal sluices from tables.

"Part IIc—Partition off raw coal surge bin.

"Part IIIa—Remove nine tables (numbered 3, 4, 5, 6, 7, 8, 21, 22, and 23) and erect No. 1 jig.

"Part IIIb—Remove present refuse elevator before erecting No. 1 refuse conveyor and cross refuse conveyor. (This required bypassing refuse to boiler coal pit and running all refuse up the boiler coal elevator.)

"Part IIIc—Erect part No. 1 of electrical control room necessary to operate No. 1 jig.

"Part IV—With No. 1 jig in operation, remove remaining old equipment, and erect No. 2 jig with remaining parts."

There were a few departures from this schedule, but in general the change-over was accomplished as planned. A comparison of the flowsheets before and after the change shows that the changes were not confined to washing machines; the whole system is involved. As of this date, no drastic changes have been made in the general operation of the fine coal system, but an investigation of more efficient operation of this part of the process is under way.

Raw Coal to Mixing Bins: The obvious change in this part of the circuit is the elimination of the Bradford breakers, screens, and roll crushers which served to reduce the 2x0 slack to approximately ⅜x0 for the tables. This has decreased the idle time at the coal dump, since it is no longer necessary to wait for the recirculating load of breaker reject to clear out between runs of different coals. The coal is now being dumped and sent directly from the receiving hopper to the appropriate bin.

During the first year operations were carried out with a mixture of a large number of coals. This was no change from the situation that had existed for 10 years, during which time it had been necessary to purchase outside coals to satisfy requirements. In mid-August 1953 it was possible through the opening of a new captive mine to eliminate the purchase of high volatile coking coal from outside mines. The mixture is much simpler now that only three mines are sending in coal of this kind.

It had become evident that the practice of using certain coals as interchangeable members of a group was causing irregularities in the feed that were automatically reflected in the product, and more accurate classification of the raw coals was anticipated. The situation is compared in Figs. 2 and 3. There is still room for improvement in mixing feed.

Mixed Raw Coal to Jigs: The raw coal bunker required for surge capacity between bins and individual tables was eliminated, and the feed now goes directly from the mixing bins to the jigs, a paddle switch controlling the air on the jigs. Any interruption of the feed automatically cuts off the air, and resumption of the feed restores the air. There is no surge bin, as such, in the present system; the mixing bins, in effect, serve a dual purpose.

The present set-up permits varying the proportion of total feed between the two jigs in any ratio desired, but to change the total feed it is necessary to go back to the mixing bins and readjust all the bin gate settings.

Since the composition of the mixed raw coal is determined by the proportion drawn from each bin, and the quantity is determined by the total gate opening,** any changes in composition or rate re-

* Contractor was the Roberts and Schaefer Co. of Chicago.

** The belts operate at constant speed.

quire that all bin gates be adjusted properly. Evidently it would have been wise at least to provide for variable speed operation of the mixing belts, with remote control from the jig floor. This would have permitted varying the load at will but retaining the desired composition.

Washing: The change in this section is so obvious as to require little explanation. Operation-wise the gain is tremendous. Flows of coal and water are much larger than formerly and far less liable to obstruction. Operation controls are centralized and it is possible to see at a glance whether or not a particular motor is still running. Weightometers indicate the input and output of the plant, and lights indicate whether or not there is a load on the mixing belts. Ammeters tell the story of load conditions at the crushers and driers, and interlocks prevent a major pile-up in case a machine fails.

Control of quality of product is simplified in many ways, as there are fewer units to watch and keep in proper adjustment.

Dewatering: All coal discharges from the jigs to flumes provided with 4x5-ft sections of ¾-mm wedge wire screen, serving as primary dewaterers. Semidewatered coal discharges to the top decks of the four Low Heads, fitted with either ⅜-in. or ¼-in. square mesh as the occasion demands. Oversize is discharged at a tolerable moisture content to the two 42x24-in. Jeffrey Flex-Tooth crushers and thence to the washed coal belt. Undersize from the top deck passes to the lower deck of 48-m equivalent wedge wire, and oversize is taken to the centrifugal driers by drag conveyor. After drying it discharges on the clean coal belt.

Undersize and water from the preliminary dewatering screens and lower decks of the Low Heads passes into two 10x0-m sumps from which it is pumped to the settling cones.

This system has one very important advantage over the old. It is virtually impossible for any wood chips, sticks, or pieces of coke to remain in the circulating water, and cleaning the screens at the equalizing tanks is now a thing of the past. It may seem unusual to include pieces of coke as unwanted flotsam in a washery water circuit, but on occasion it is necessary to pick up *hot coal* from storage, and some of this is already coked before it can be washed. It is, perhaps, superfluous to mention that when a high percentage of reload coal is used conditions are not what they are without it.

Another interesting point is that for the first time it is possible to get the washed coal too dry, in that capacity is lost at the ovens if all coal is dried as much as possible. There also appears to be a tendency for *dry coal* to cause excessive carbon deposits in the ovens. Experience at the Rosita plant of the American Smelting and Refining Co. at Chihuahua, Mexico, also indicated that this treatment of washed coal could result in its being too dry. The solution to this problem is to bypass some of the material from the lower deck of a Low Head around the driers.

Crushing: The new flowsheet, Fig. 3, provides for crushing the coal after it is washed. It was intended that pulverization of washed coal under the old system be matched by crushing of +¼-in. material after dewatering. Current operations indicate that it will be possible to maintain between 70 and 75 pct −⅛-in., with only a small amount of +½-in., in the total washed coal.

Fine Coal Recovery: On the surface this section appears to have been left unchanged. Fine coal and water are still pumped to the settling cones, settled solids are taken to the vibrating filters, a certain tonnage of fine coal is recovered as vibrator cake, and the remainder of the coal and water is recirculated. However, there have been changes in size and quantities of solids, as well as general closing up of the system, and studies are in process to determine what revisions of method or equipment are required to bring this section into line with the rest of the operation.

At the time the new washery was being planned a decision on what to do about fine coal and recirculating water was purposely delayed. It was believed that by the time the rest of the plant was operating at or near ultimate capacity it would be possible to make a much better estimate of requirements. This now appears to have been even wiser than was originally thought. Owing in large part to mechanized control of the water level in the cones and pump sump, losses of fine coal are considerably lower than formerly. The only losses are very fine high ash solids in overflow from the Dorr thickener and effluent from the Bird polisher, which operates on thickener underflow.

Refuse: Refuse had been hauled away in railroad cars for many years, but the increased traffic around the washery after the 2000 tons per shift was passed necessitated haulage by truck. This change has resulted in a saving of direct labor at the washery, since the driver loads his own truck, and the charge for the trucking is much less than the previous railroad charges.

Washed Coal: No change was made in the washed coal conveyor, except to install a weightometer and

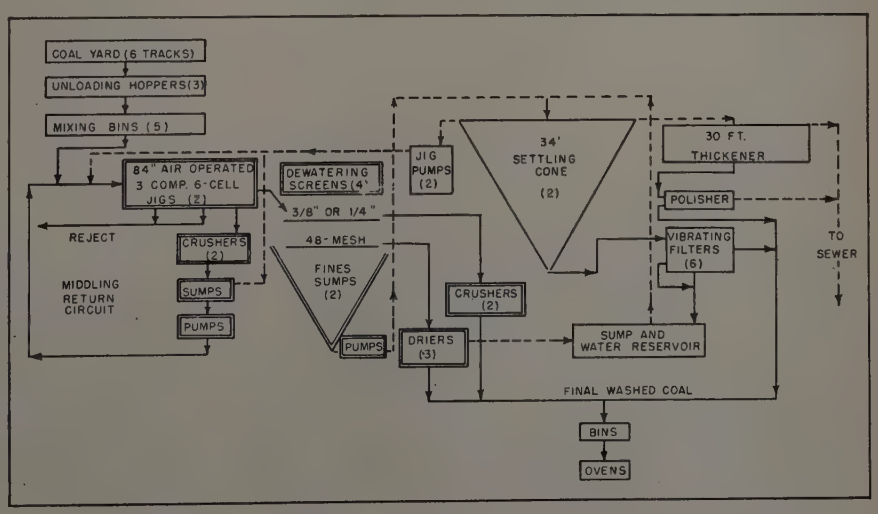


Fig. 3—In this latest flowsheet the machines in double lines are new. Dotted lines represent flow of water or thin slurries. Solid lines represent flow of coal or other solids or sludges.

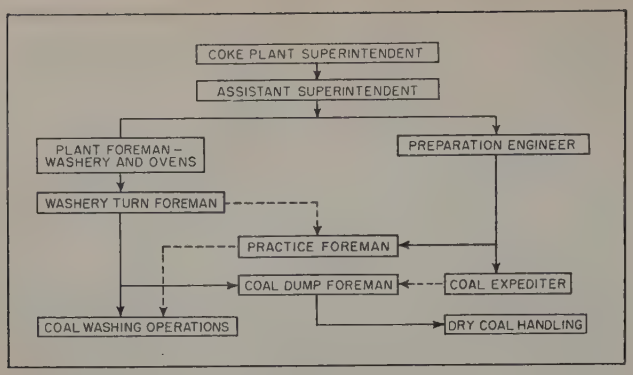


Fig. 4—Manpower requirements shown here should be compared with those in Table I.

put the starting switch on the central control panel. An indicating light shows whether clean coal is going to the main storage bin or is being bypassed direct to the oven bins.

Overall Operation: It should be evident that plant operation is now a great deal simpler than before the change. A multitude of small units operating in parallel have been eliminated, and controls for the entire plant have been consolidated.

On the other hand, there is more danger of losing a large percentage of throughput in case of a breakdown at the jigs. In an emergency, however, one jig can handle as much coal as the old table plant operating at full capacity.

Labor Requirements

Fig. 4 indicates the reduction of manpower required per operating shift. Since output has increased from about 1650 tons per shift to about 2650 tons, manhours of labor are half the previous figure. Fig. 4 illustrates the flow of authority from top level to operating force. Solid lines indicate direct supervision and dotted lines functional supervision. At the time this reorganization was effected, certain

Table I. Promotional Line at Pueblo Coal Washery Before and After Reorganization

March 1949		March 1953
Group A	**Group B**	
1 Table operator	1 Head coal unloader	1 Coal expediter
2 Vibrator attendant	2 Car spotter	2 Jig attendant
3 Handyman	3 Car spotter helper	3 Fine coal man
4 Assistant table operator	4 Coal unloader helper	4 Handyman (coal unloader)
5 Washed coal conveyor man		5 Washery oiler
6 Crusherman		6 Conveyor man
7 Oiler		7 K&X conveyor man
8 Drierman	**Group C**	8 Mixing bin man
9 Water tender	1 Table repairman	9 Washery helper
10 Tripper man	2 Table repairman helper	10 Cleanup helper
11 K&X conveyor man		11 Labor
12 Mixing bin man		
13 Crusher operator helper		
14 Cleanup helper		
15 Labor		

government regulations interfered with plans to make the coal expediter a company man on salary, whose experience in this position would train him to become practice foreman. Consequently the coal expediter is top man in the operating line-up, and present incumbents were selected from the seniority list after a considerable period of weeding out those not capable of handling the job.

Weekly or oftener, if necessary, the coal expediter prepares detailed schedules for each operating shift, based on need for the various coals and estimated availability as determined from schedules of operations at the mines and records of shipment and receipt of individual cars.

The practice foreman, in charge of the actual washing operation, is primarily concerned with quality of product. Since quality cannot be maintained unless operations are smooth and continuous, he is interested in the condition of the machinery and makes frequent inspection tours. He is responsible for making the necessary adjustments in air pressure, water, and float counterweights to suit the mixture being washed and keeps a log book in which he reports operating conditions and difficulties. Control of the mixture over longer and longer periods of time has contributed materially to the improvement of operations.

Table I indicates streamlining of the operating force before and after the reduction in manpower.

Washery maintenance is handled by the mechanical department of the steelworks. There are three teams of millwrights and helpers, one for the fine coal system, one for the remainder of the washery, and one in the dry coal handling system. It is too early to state that repair and maintenance is either more or less expensive than formerly. The cost sheets, when interpreted by one familiar with the changes made in the flowsheet, indicate that after a time reduction in these costs may well be expected.

At the start there was considerable trouble with the pumps, but after installation of a separate high-pressure line for gland water there has been much less difficulty.

The air intake for the jig blowers is located on the side of the washery next the coke ovens. Fine coke dust appeared to be causing trouble in the air valves. This was remedied by installation of large oil bath air cleaners.

Live steam is available for cleaning the float mechanisms at the main jig floor level and the ejector mechanisms at the lower jig floor level. Cleaning and inspection have been facilitated by placing windows of 2-in. mesh welded wire fabric in some of the guards. This fabric has also been used to guard the tops of drag conveyors.

Except as used for sample preparation, hammermills are new to the staff. The first difficulties with these machines were due to poor design of the discharge chutes, which tapered in from all four sides, causing clogging of the mill from the bottom. Now the chutes have been altered this no longer occurs.

The present centrifugal driers are requiring less manpower for maintenance than the old driers, and

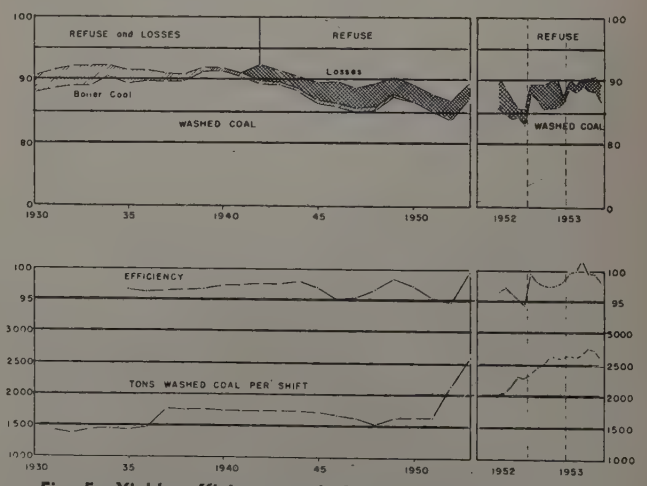


Fig. 5—Yields, efficiency, and throughput. Probably no attempt will be made to increase hourly tonnage, as auxiliary equipment is nearing the point of diminishing returns.

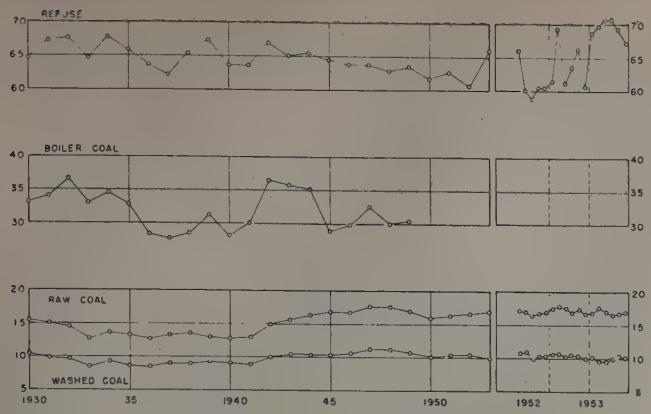


Fig. 6—Percent ash, raw coal, and products. Boiler coal is no longer used, but comparative statistics are of interest.

a much smaller unit handles as much material or more. It was necessary to take drastic steps in handling the fine coal. Some of the settling cone underflow has been put in the driers whenever the circulating water has been too heavy. It has been demonstrated that these driers can handle this type of material, but it is not at all certain that they should be expected to do it indefinitely. The addition of this type of material to the normal feed results in excessive fluctuations in the load at the motor, which probably is a very mild reflection of the strains on the internal gearing. Apparently there is a lower limit to the size of material which should be fed these machines if long, trouble-free life is expected from them.

Comparative Statistics

Statistics of these endeavors have justified most of the expectations for the new plant. Figs. 5 and 6 illustrate in part the extent of improvement to date. To illustrate the general trend from 1930 to 1953 yearly averages are given, inclusive of all items. On the extreme right of each chart a panel shows monthly averages from August 1952 through December 1953. Predictions are based on the assumption that the mixture can be controlled as well in the future as in the last six months.

Fig. 5. Tonnage, Efficiency, and Yields: Throughput per 8-hr shift has definitely been boosted, reaching a new high in efficiency and minimizing losses. It is unlikely that an attempt will be made to increase hourly tonnage appreciably, for indications are that all auxiliary equipment is nearing the point of diminishing returns. Probably the jigs themselves could handle a considerable increase in tonnage if they were not held back by the rest of the equipment, for one jig will do good work on a load considerably greater than half the total now fed to two jigs operating simultaneously.

Although efficiency cannot be boosted very much, it will be maintained at the current level, or slightly better.

$$\text{Efficiency} = \frac{100 \times \text{tons } \textit{dry} \text{ washed coal}}{\text{tons } \textit{dry float in feed} \text{ at W.C. ash}}$$

Losses indicated on the chart are 1—moisture loss and 2—loss unaccounted for. Moisture loss usually amounts to between 1.0 and 1.5 pct and is due to correction of washed coal to 2.5 pct moisture; raw coal contains 3.5 to 4.0 pct moisture. Losses unaccounted for may or may not be entirely real, since they are actually a balancing figure and on occasion have been negative.

Fig. 6. Ash Content of Raw Coal and Products: Since 1950 raw coal ash appears to have increased slightly, but in the past 18 months washed coal ash has been maintained at the same level.

Boiler coal is now a thing of the past, and this data is included merely as a matter of interest. Note that it was possible to get by with a fairly high ash content for three years, 1942 through 1944, but the power department later refused to burn solid fuels.

Refuse ash had gradually declined over almost the entire period since 1930. This is largely explained by the fact that an attempt was being made to keep the washed coal ash from going up as rapidly as the raw coal ash. Chart fluctuations for refuse ash in the last 18 months can be attributed partly to conditions of which the operating personnel were unaware and partly to inexperience.

The major problem is common to all wet washing plants: control of circulating water density and complete recovery of fine coal. Eventually this will be complicated by the necessity for keeping even the undesirable fines out of the streams. Since there is a large amount of semicolloidal clay in the water this may become a big difficulty, for it is not possible to operate on a closed water circuit for more than a few hours at a time. It would be disheartening to have to remove clay from water, at considerable expense, and then haul it away.

Better results are achieved with *thin* water, that is, the specific gravity should not be allowed to go above 1.04 to 1.05 and probably should be 1.03 to 1.04. It is certain that densities higher than 1.05 are not desirable and around 1.08 are impossible.

The mechanization of water tending and reduction of the amount of water in circulation have helped considerably, but simple gravity settling of the fine coal in the present settling cones does not appear to be sufficiently rapid to accomplish what is necessary. Total area of the settling cones is about 1800 sq ft, and these were installed for use in a plant with approximately half the present capacity. The original design was good, for there is no difficulty at all if only one jig is used.

Experiments with a large cyclone thickener are now under way. It is hoped that the cyclone will thicken the vibrator to the point where it will not be necessary to maintain an overflow there. If this can be done, there will be an appreciable reduction in the circulation of water through the cones, and if this is great enough it may be possible to maintain the desired specific gravity of the circulating water without undue expense.

When water and coal mixture problems are under control simultaneously serious consideration can then be given minor problems such as determining the proper air valve settings, the proportions of *over* and *under* water, and the number of pulsations per minute.

Summary

Certain goals have been reached. Washed coal of acceptable quality is being produced with greater efficiency than ever before and with about half the labor per ton. Control of coal supply has been brought to the point where the plant can operate on a much more stable mixture and with lower railroad charges for demurrage, both of which contribute materially to reducing washing costs. Once the fine coal problem is under control, techniques can be refined to the point where everything possible is being achieved with the available material and equipment.

Adsorption of Dodecylammonium Acetate On Hematite and Its Flotation Effect

by A. M. Gaudin and J. G. Morrow

FLOTATION requires the existence of a definite contact angle. This contact angle, the surface tension of the solution, and adsorption at the solid-fluid interface are quantitatively related.

Adsorption of dodecylammonium acetate on hematite was measured for a wide range of concentrations of reagent in solution. Similar measurements for quartz have already been made.[1] Contact angle measurements were then made on polished surfaces of hematite and of quartz immersed in aqueous dodecylammonium acetate solutions, and a functional relationship was sought between adsorption density at the mineral-solution interface and the contact angle. Finally, the surface tension of the aqueous amine acetate solutions was measured. These data were combined to give an evaluation of the work of adhesion for the three-phase system.

Specular hematite was crushed and then ground dry in a laboratory porcelain mill, with flint pebbles, to pass a 200-mesh screen. The ground product was sized in a Haultain infrasizer, and one of the granular sizes (cone No. 3) was used in all adsorption tests. A hand magnet was used to remove magnetite and abraded iron. Quartz was removed in a Frantz isodynamic magnetic separator. The purified hematite was leached in aqua regia, washed with distilled water until the washings appeared free of electrolyte by conductance measurement, dried in a low-temperature oven, and stored in a pyrex container.

The specific surface of the closely sized hematite was determined by the krypton gas adsorption method.[2] Three measurements gave an average value of 1350 sq cm per g. Chemical analysis showed Fe = 69.37 pct, insol = 0.72 pct.

The quartz used in the flotation tests had been prepared by Chang[3] for an earlier investigation.

Demineralized distilled water was used for all test solutions. Dissolved salt content was of the order of 0.03 ppm, expressed in terms of sodium chloride, as estimated from conductance measurements.

Dodecylammonium acetate was obtained from Armour & Co.[4] in two forms, the unmarked compound and a preparation marked by carbon 14 in the hydrocarbon chain of the aminium ion. Specific activity of the active salt was 0.134 millicurie per g.

The important physico-chemical constants for the primary amine salt have been reviewed by de Bruyn.[1] The calculated effect of hydrolysis of amine salt on pH of aqueous solutions and the effect of pH on the distribution ratio of the alkylammonium ion to free amine are of particular interest.

All other chemicals used in this investigation were of analytical reagent grade. A *column method*[1] was used for adsorption work.

Attainment of equilibrium distribution in the adsorption column depends on the solid-solution contact time, hence upon the volume of solution passed. It was assumed that contact time required for equilibrium would be a maximum for the lowest reagent concentrations. On this premise it was demonstrated experimentally that the passage of 500 ml of solution through the mineral bed was adequate.

An aliquot (1 to 5 cc) of the solution to be analyzed was transferred to a small pyrex cup and allowed to evaporate to dryness at room temperature; 4 or 5 mg of unmarked amine acetate were added to the dried sample and the cup and its contents were transferred to the combustion system for analysis. Evaporation at room temperature must be emphasized, as even slightly elevated temperatures result in loss of reagent. A laboratory model G Beckman pH meter equipped with a glass electrode was used to measure pH of amine acetate solutions.

The technique of internal gas counting of radioactive carbon dioxide in a Geiger-Muller counter was used. Developed originally by Brown and Miller,[5] this method was adapted to the analysis of carbon-14 marked flotation reagents by Chang, de Bruyn, and Bloecher.[6] Analytical method and procedures have been described in detail by Bloecher.[7]

Contact angles were measured by the captive-bubble technique.[8] The mineral specimens were carefully selected to avoid cracks and inclusions of other minerals. All specimens were mounted in plastic and polished to produce a smooth surface. The final polishing and contact-angle measuring

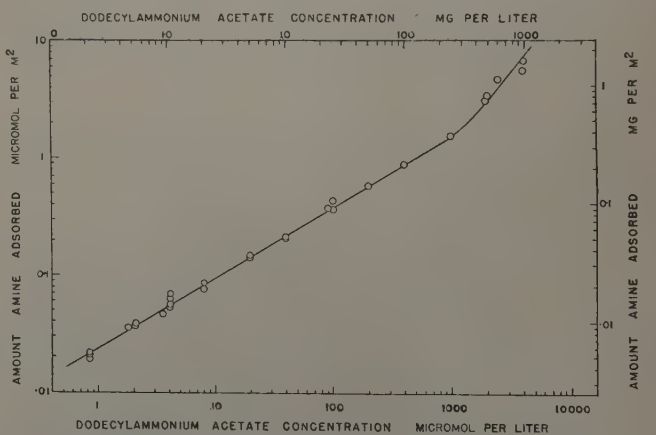


Fig. 1—Adsorption of dodecylammonium acetate on hematite as a function of concentration.

A. M. GAUDIN, Member AIME, is Professor of Mineral Engineering, Massachusetts Institute of Technology. J. G. MORROW, Student Associate AIME, is Research Metallurgist, Freeport Sulphur Co., Freeport, Tex.

Discussion on this paper, TP 3932B, may be sent (2 copies) to AIME before Feb. 28, 1955.

Manuscript, April 8, 1954. Abstracted from the thesis by J. G. Morrow submitted in partial fulfillment of the requirements for the degree of Doctor of Science at Massachusetts Institute of Technology. Chicago Meeting, February 1955.

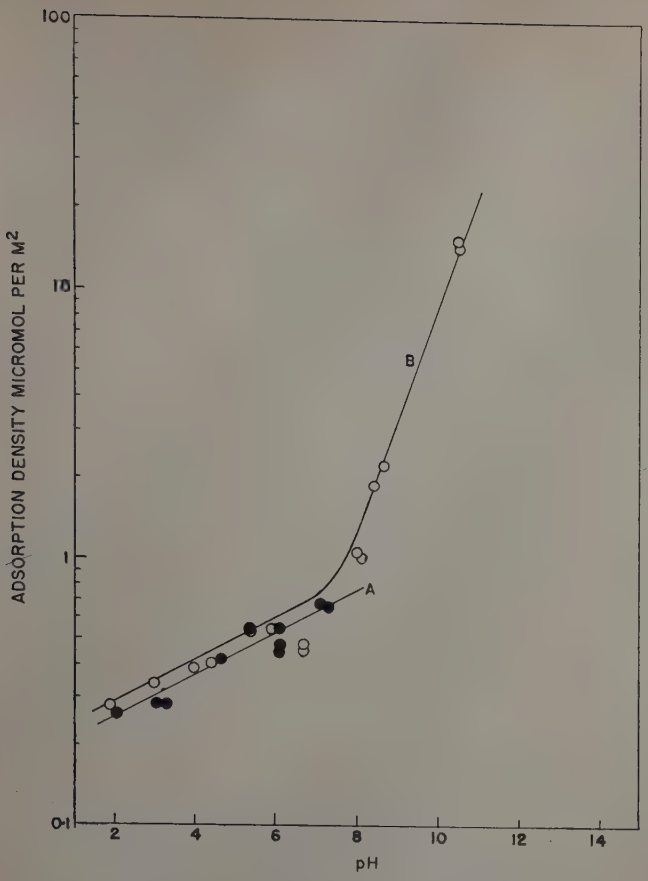


Fig. 2—Adsorption of dodecylammonuim acetate on hematite (*A*) and quartz (*B*) as a function of pH. (Data for quartz according to unpublished data by de Bruyn.)

techniques described by del Guidice[9] were used. Conditioning of the polished surfaces in amine solution was done in the optical-glass cell with a mechanical stirrer. The conditioning time was ascertained by trial to give the maximum angle at each concentration.

A Cenco-duNouy tensiometer was used to measure the surface tension of aqueous solutions of dodecylammonium acetate at 25°C. The concentration of dodecylammonium acetate required to induce incipient flotation of hematite and quartz was determined by the vacuum-flotation technique of Schuhmann and Prakash.[10]

Adsorption Results

The Adsorption Isotherm: The amount of agent adsorbed on hematite per unit area of solid (Γ) from aqueous solutions of dodecylammonium acetate was measured at concentrations ranging from 0.8 to 4080 micromol per liter. Results are given in Fig. 1, in which log Γ is plotted against log c. Adsorption is expressed in milligrams per m^2, or in micromols per m^2, the latter being numerically equal to gibbs.[11] Minor variations in temperature occurred during the tests so that reference to Fig. 1 as an *isotherm* is not rigorously correct. All pH readings (5.8 to 6.6) indicated slight acidity. A trend toward higher pH values was noticeable with increasing concentrations of the agent.

Fig. 1 shows that adsorption density increases with increasing concentration. Over the concentration range from 0.8 to about 1000 micromol per liter log Γ is proportional to 0.6 log c. This means that Γ increases approximately as $c^{0.6}$. At higher concentrations Γ increases more rapidly with c. The bend in the isotherm at about 1000 micromol per liter occurs at a surface coverage about one quarter of a complete monolayer. The area required per ion adsorbed was assumed to correspond to two oxygen sites, or 24.08 A^2 of surface.[12, 13] It is assumed further that the adsorbate attaches itself to the mineral surface through its polar end with the hydrocarbon chain oriented perpendicular to the mineral surface. On these premises a monolayer will be obtained for an adsorption density of 6.90 gibbs.

Effect of pH on Adsorption Density: The adsorbate concentration in the test solution was maintained at approximately 100 micromol per liter (25 mg per liter) while the pH was varied from 2.1 to 7.2 with HCl or NaOH. Adsorption tests made in more alkaline solutions were discarded because the solutions became turbid. Results obtained are presented in Fig. 2. A variation in pH on the acid side of neutrality has only a small effect on adsorption density. In fact, for a ten thousandfold change in hydrogen ion concentration the corresponding change in Γ is only twofold. The effect of pH on the adsorption of amine on quartz[1] (40 micromols per liter) is also shown in Fig. 2. Here adsorption varies only slightly with pH in acid solutions but increases rapidly in alkaline solutions.

It was experimentally ascertained that the adsorption density is independent of the specific surface of the hematite.

Reversibility of Adsorption and of Desorption: A series of 10 tests was made to determine the exchangeability between adsorbed agent and agent in solution. Hematite was first coated with inactive dodecylammonium acetate by passing 500 ml of amine salt solution through the mineral column. Subsequently a known volume of radioactive amine acetate solution of the same concentration was passed through the pre-coated mineral bed. The observed adsorption determined by radio-assay represented the amount of radioactive amine from solution that had exchanged with unmarked agent

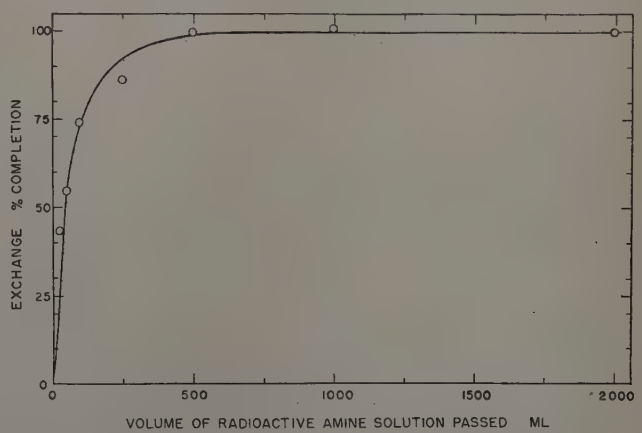


Fig. 3—Reversibility of adsorption of dodecylammonium acetate on hematite. Radioactively marked agent replaces the inert agent.

adsorbed at the mineral surface during the precoating treatment. The adsorption expected for complete exchange was determined from two tests in which only the active amine solution was passed.

Results are shown in Fig. 3. Complete exchange between the unmarked and radioactive amine occurred when volumes of 500 ml or more of the radioactive amine solution were passed through the pre-coated mineral bed. Under conditions prevailing in these tests the passing of smaller volumes was inadequate for complete exchange.

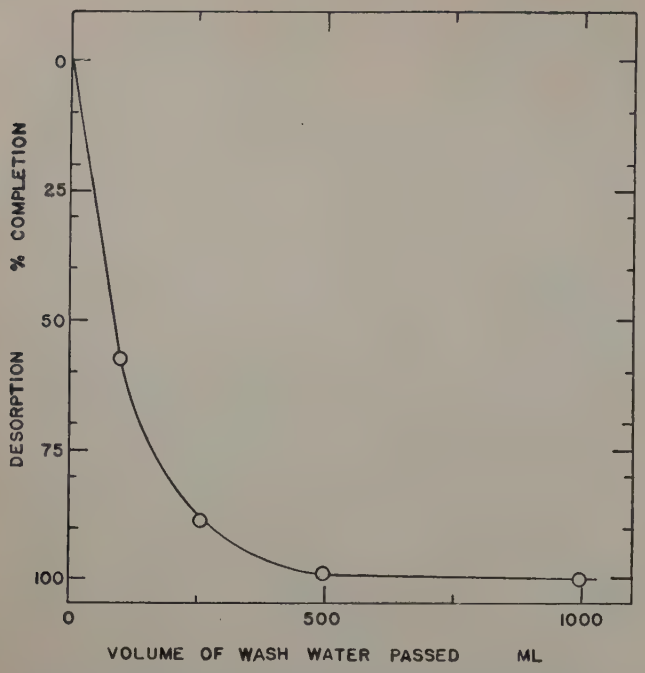


Fig. 4—Reversibility of adsorption of dodecylammonium acetate on hematite. Water leaching of adsorbed marked agent.

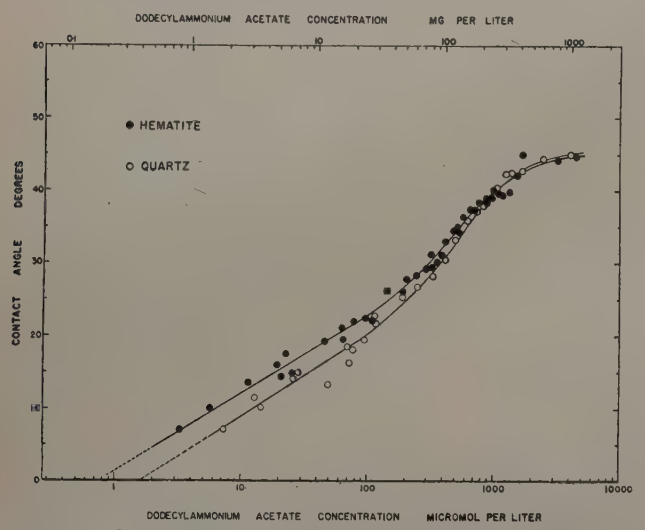


Fig. 5—Contact angle on hematite and quartz in solutions of dodecylammonium acetate as a function of concentration.

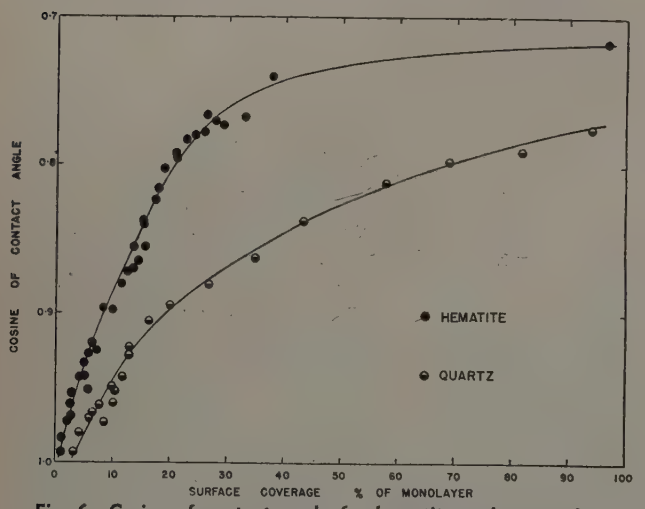


Fig. 6—Cosine of contact angle for hematite and quartz in solutions of dodecylammonium acetate as a function of the surface coverage of the solid surface by the adsorbed agent. Constant pH conditions.

In another series of tests the mineral was first pre-coated with radioactive amine acetate by passing through the mineral bed 500 ml of solution containing 204 micromol per liter of the agent. Then a measured volume of water was passed through the same bed. The difference between the equilibrium amount of adsorption and the amount remaining on the solid after the water wash gave a measure of the desorption or leachability of the adsorbed amine.

Fig. 4 shows a plot of desorption, expressed as percent completion, vs volume of water passed through the mineral bed. It is obvious that the reagent is readily washed off the mineral surface.

Contact Angle Results

Contact angle measurements were made on polished surfaces of both polycrystalline and monocrystalline hematite in aqueous amine acetate solutions. Conditions of essentially constant pH (6 to 7) and temperature were maintained. Fig. 5 is a plot of the contact angle, expressed in degress, vs the logarithm of the concentration of amine acetate in solution. Fig. 6 shows the relationship between the cosine of the contact angle and the surface coverage.

Fig. 5 suggests that a measurable adsorption density is required before a finite contact angle is obtained. The angle increases slowly with increasing concentration until at 1 millimol per liter (250 mg per liter) of agent in solution a contact angle of 38° to 40° is obtained. Further increases in the concentration have a small effect on the magnitude of the angle, whereas the adsorption density increases rapidly, see Fig. 1. An apparent maximum angle of 45° is indicated. In terms of the surface coverage it is interesting to note that 20 to 25 pct surface coverage gives an angle of about 38°, while at nearly complete coverage the contact angle is only 45°. It will be recalled that the bend in the adsorption isotherm (Fig. 1) occurred at approximately 25 pct surface coverage.

A number of measurements were made in which the polished surface was conditioned in the amine acetate solution and then transferred to a bubble cell. The contact angle was then measured in water. In each case the contact angle diminished to such a small value it could not be measured. This is not surprising in the light of the data that have already been presented showing the ease with which the adsorbate can be washed off the mineral surface.

Measurements of contact angles on polished surfaces of quartz are also included in Figs. 5 and 6. For Fig. 6 de Bruyn's[1] data on quartz adsorption were used. Fig. 5 indicates that the contact angle on quartz increases with increasing concentration in about the same way as the angle increases in hematite. At low concentrations the angle on hematite is somewhat larger than the angle on quartz for any given concentration. This difference diminishes with increasing concentration. Here, also, a maximum angle of 45° is indicated. At approximately 280 micromol per liter (70 mg per liter), the concentration corresponding to the bend in de Bruyn's adsorption isotherm on quartz, the contact angle is about 28°. The amount of amine acetate adsorbed at 280 micromol per liter (70 mg per liter) is 22 pct of that required to form a monolayer.

Quartz surfaces conditioned in amine solutions and then transferred to the bubble cell filled with water exhibited only cling angles after a short conditioning period.

Effect of pH on the Contact Angles of Hematite and Quartz at Constant Concentrations of Amine

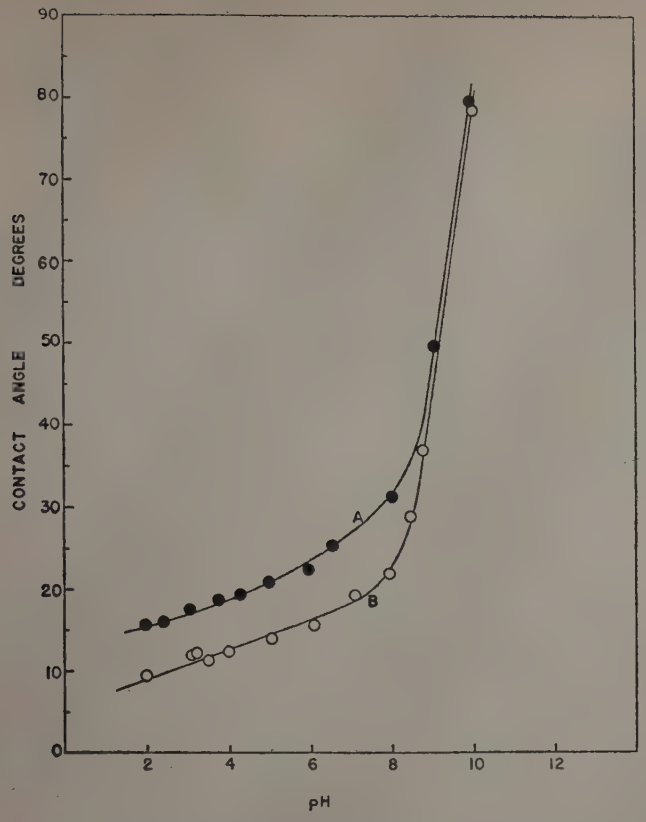


Fig. 7—Contact angle on hematite (*A*) and quartz (*B*) in solutions of dodecylammonium acetate (102 and 40.8 micromol per liter, respectively) as a function of pH.

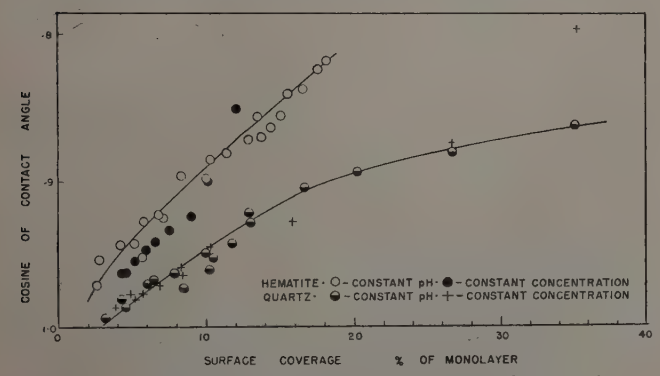

COSINE OF CONTACT ANGLE
HEMATITE · ○ - CONSTANT pH · ● - CONSTANT CONCENTRATION
QUARTZ · ◒ - CONSTANT pH · + - CONSTANT CONCENTRATION
SURFACE COVERAGE % OF MONOLAYER

Fig. 8—Cosine of contact angle for hematite and quartz in solutions of dodecylammonium acetate as a function of surface coverage. Constant pH or constant concentration of agent in solution.

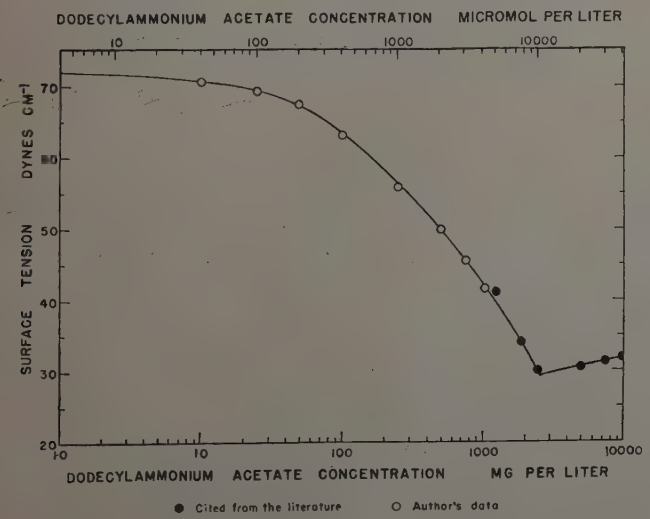

DODECYLAMMONIUM ACETATE CONCENTRATION MICROMOL PER LITER
SURFACE TENSION DYNES CM⁻¹
DODECYLAMMONIUM ACETATE CONCENTRATION MG PER LITER
● Cited from the literature ○ Author's data

Fig. 9—Surface tension of aqueous dodecylammonium acetate solutions, in function of concentration.

Acetate in Solution: Contact angles were measured on polished surfaces of hematite and quartz in aqueous amine acetate solutions in which the concentration of the salt was maintained constant while the pH was varied from 2 to 10. Concentrations of 102 and 40.8 micromol per liter were used for hematite and quartz respectively. The pH was regulated with hydrochloric acid or sodium hydroxide.

Fig. 7 is a plot of contact angle vs pH. The angle increases slowly with increasing pH up to a pH value of about 8. Thereafter even a slight increase in pH results in a large increase in the contact angle.

In Fig. 8 the coordinates are the same as in Fig. 6, that is, the cosine of the contact angle is plotted against percent surface coverage. In this figure data for each mineral are given in terms of measurements made at substantially constant pH and variable concentration on one hand, or at variable pH and constant concentration on the other. It seems that within the range covered by Fig. 8 the contact angle is related principally to the adsorption density rather than to the concentration of the pH.

Surface tension data for aqueous amine acetate solutions (pH near 6) measured at various concentrations and at room temperature, 25°C, using the ring method are presented graphically in Fig. 9. Solid circles show values from the literature.

Fig. 9, a plot of surface tension vs the logarithm of the concentration of amine acetate in solution, is a typical surface tension-concentration curve. It shows at first only a gradual decrease in surface tension with increasing concentration, then a more rapid decrease until a minimum value is reached. Thereafter, a slight increase is observed with increasing concentration. The minimum value of the surface tension is about 30 dynes cm^{-1} and occurs at a concentration of 1.2×10^{-2} mols per liter, essentially that of the critical micelle concentration (1.42×10^{-2} mols per liter).[16]

The work of adhesion of an air bubble to a particle may be given by

$$W = \gamma_{LA} (1 - \cos \theta) \qquad [1]$$

in which γ_{LA} is the surface tension of the liquid and θ the contact angle.[17]

Fig. 10 shows the calculated values of W plotted against Γ for hematite and quartz. Each curve shows W to increase and pass through a very flat maximum. Thereafter W appears to decrease slightly with increasing surface coverage. The maximum values for hematite and quartz are 13.5 and 13 erg per cm^2 respectively and occur at surface coverages of about 0.35 and 1.05 parts of a monolayer respectively. The values of W corresponding to the conditions necessary for good flotation of hematite and quartz, namely, 4 to 5 pct surface coverage, are of the order of 2.5 and 5 erg per cm^2 respectively.

The maxima in these curves are not well defined. Quite possibly the curves are more nearly horizontal to the right of the so-called maxima. Additional measurements of higher precision are required.

Energy of Adsorption: Eq. 1 may be regarded as an expression of the total work of adhesion corresponding to a given adsorption density, Γ. Therefore the increment, X, in the work of adhesion, for unit change in Γ is given by

$$X = \frac{dW}{d\Gamma} = \frac{d[\gamma(1 - \cos \theta)]}{d\Gamma} \qquad [2]$$

This quantity is the slope of the curve relating work of adhesion to adsorption density, that is, the slope

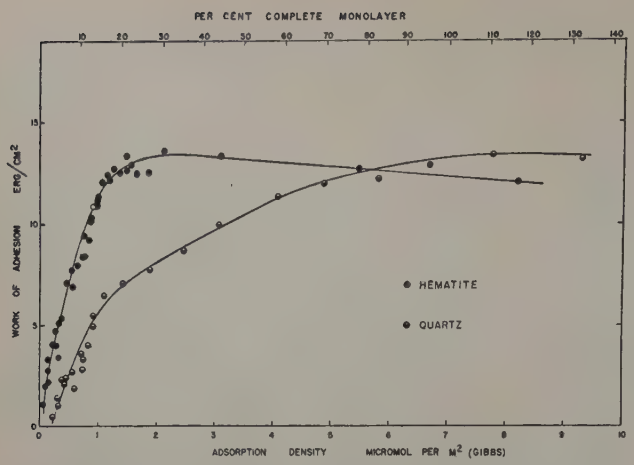


Fig. 10—Work of adhesion of hematite and quartz to air from aqueous solutions of dodecylammonium acetate, in function of surface coverage.

of the curves in Fig. 10. X may be thought of as the energy of adsorption of the latest adsorbate to be adsorbed.

Typical values of X for various values of Γ are given in Table I. For small surface coverages X is quite large. With increasing surface coverage, however, X decreases and actually changes in sign at high adsorption densities.

If the first ions to be adsorbed occupy the most active sites on the surface and are randomly situated, and if subsequently adsorbed ions occupy less and less active sites and remain independent of their neighbors on the surface, then the change expected in adsorption energy with changing adsorption density should be similar to that shown in Table I. The last ions that could be adsorbed and still remain independent of their neighbors would not be expected to have a large energy of adsorption. Hence the occurrence of low values for X at high concentrations is not surprising. However, a negative value for X is difficult to rationalize.

Correlation of Results and Interpretation

The authors' adsorption isotherm for hematite, see Fig. 1, and de Bruyn's isotherm for quartz[1] are remarkably similar. Adsorption densities (Γ) increase with increasing concentration (c) at low reagent concentrations. Similar breaks occur at intermediate concentrations. At high concentrations Γ increases more rapidly than c.

At low concentrations the log Γ-log c curve approaches a straight line of slope 0.5 in the case of quartz and 0.6 in the case of hematite.

Empirically it is possible to find the parameters for an equation

$$\Gamma = Ac^{0.5} + Bc \qquad [3]$$

which fits the data for hematite over the entire range studied. Such a curve is shown in Fig. 11, together with the experimental points. The values of A and B in Eq. 3 are 2.02×10^{-9} and 1.45×10^{-7} respectively.

This empirical result was found by assuming that the Bc term may be neglected in comparison with the $Ac^{0.5}$ term for the lowest experimental concentration ($c_1 = 2.45 \times 10^{-7}$ mol per liter) and by testing various B values until a satisfactory fit was obtained.

It is clear that the curves of Fig. 9 and Fig. 1 are alternative ways of presenting the data. In Fig. 1 two mechanisms are involved of which one appears at a higher concentration only, while in Fig. 11 a twofold mechanism is implied as occurring across the concentration range studied. The authors are unable to choose between these interpretations of the data, but it may be profitable to discuss briefly the implications of these diagrams.

De Bruyn concluded from his study of the adsorption of dodecylammonium acetate on quartz that neither a stoichiometric ion-exchange mechanism between the hydrogen ion and the aminium ion nor the adsorption of free amine is in harmony

Table I. Energy of Adsorption (Change in Work of Adhesion with Change in Adsorption Density)

Solution Concentration		Quartz			
Mg Per Liter	Micromol Per Liter	Adsorption Density, Pct of Monolayer	Surface Tension, Erg Per Cm^{-2}	Contact Angle, °	X Calorie Per Mol
0.8	3.2	2	72.2	4	
4.3	17.5	5	71.3	12	2690
17.2	70	10	70.2	20	1460
80	318	25	64.9	28	504
135	550	50	61.4	34	301
240	980	100	56.7	40	97
292	1190	125	55	42	−20
			Hematite		
4.6	18.8	2	71.3	15	3830
21	86	5	69.9	22	2920
66	268	10	66.2	29	1985
132	540	15	61.5	35	1410
252	1030	25	56.3	40	240
550	2250	50	48.9	44	−60
980	4000	100	42.5	45	−60

with experimental results. He favored an ionic adsorption process involving formation of an electrical double layer at the surface.

Consideration of the formation of an electrical double layer appears sound, since electrokinetic measurements have shown that particles dispersed in distilled water carry an electric charge.[18] The system as a whole must be electrically neutral; consequently each particle must be surrounded by an electric double layer. The negative surface charge on quartz is attributable to specifically adsorbed hydroxyl ions. This charge is balanced by an equivalent charge of the opposite sign which is carried by ions (generally called *gegen* ions) in a diffuse layer extending from the adsorbed layer into the interior of the liquid phase. It may be thought that ferric oxide would be qualitatively similar to silicon oxide.

The simplest model of the electrical double layer is that of Gouy and Chapman.[18] The surface is con-

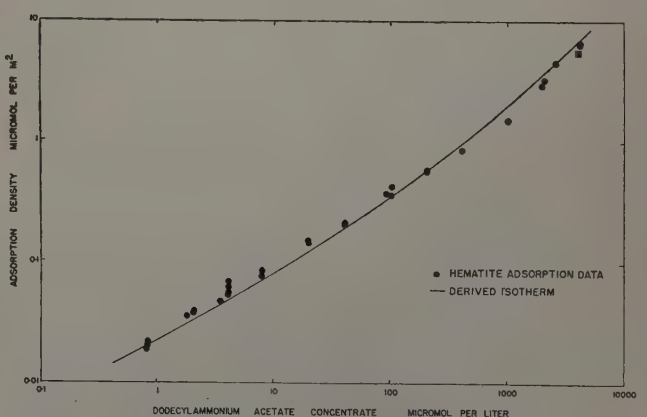

ADSORPTION DENSITY MICROMOL PER M²
HEMATITE ADSORPTION DATA
DERIVED ISOTHERM
DODECYLAMMONIUM ACETATE CONCENTRATE MICROMOL PER LITER

Fig. 11—Adsorption of dodecylammonium acetate on hematite fitted to an equation of the form $\Gamma = Ac^{0.5} + Bc$ with $A = 2.02 \times 10^{-9}$ and $B = 1.45 \times 10^{-7}$.

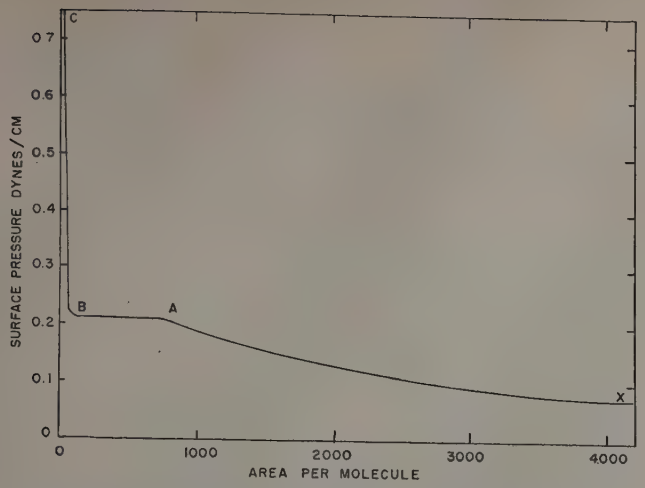


Fig. 12—Surface pressure vs area per molecule for myristic acid adsorbed on water at 14.5°C according to N. K. Adam, *The Physics and Chemistry of Surfaces.*[17]

sidered as a smooth plane with an homogeneous surface charge. The diffuse layer contains both positive and negative ions as the electrostatic attraction of the negative surface charge on the cations is counteracted by the thermal motion of these ions, which tends to equalize the distribution of ions throughout the liquid. The surface charge per unit area (σ_s) is numerically equal to the charge per unit cross section of the diffuse layer (σ_d).

The relation between the diffuse layer charge (σ_d), the potential drop across the diffuse layer (ψ_o), and the total electrolyte concentration (n ions per cc) is given by the Gouy-Chapman equation:

$$\sigma_d = -\sqrt{\frac{2\epsilon kT}{\pi}}\sqrt{n}\,sinh\left(\frac{Ze\psi_o}{2kT}\right). \qquad [4]$$

In this equation ϵ is the dielectric constant of the liquid medium, k is the Boltzmann constant, T is the absolute temperature, e is the electronic charge, z is the valency of the gegen-ions, and *sinh* denotes the hyperbolic sine.

In the case of hematite, if it is accepted that the hydroxyl ion is the potential-determining ion, then at constant pH, ψ_o is constant, and Eq. 5

$$\sigma_d = A\sqrt{n} \qquad [5]$$

may be accepted as an approximation. Since the amine salt is almost completely ionized in the vicinity of pH6, and at great dilution, and since the amine salt is the only electrolyte added, Eq. 5 can be replaced by Eq. 6, which relates the charge per unit cross-section of the diffuse layer σ_d to the concentration of amine salt added, A being a constant:

$$\Gamma_{RNH_3^+} = \sigma_d = A\sqrt{c} \qquad [6]$$

If in addition to the gegen-ions the surface of the hematite has adsorbed either free amine or a reaction product, in other words, if it is postulated without specification of a definite reaction mechanism that

$$\Gamma_{TOTAL} = \Gamma_{RNH_3^+} + \Gamma_m \qquad [7]$$

an equation of the form of Eq. 3 may be obtained provided Γ_m is proportional to c. The latter condition is fulfilled in Langmuirian adsorption at high dilution.

This rationalization of the data which makes them fit Eq. 3 implies then that at all concentrations the adsorption is in two forms, one ionic at a relatively large distance from the surface as the diffuse part of a double layer, and the other of unspecified form located very near the surface.

A different rationalization of the data may also be defended. It is well known that the adsorption-surface tension data for many surface-active molecules on water, e.g., myristic acid, $C_{13}H_{27}COOH$, may be represented in the form of a diagram like Fig. 12, in which the reciprocal of the adsorption is plotted as abscissae and the surface pressure as ordinates. As the adsorption increases, the surface pressure increases; this is represented by the portion XA of the curve. At the area per molecule represented by A a new condition sets in represented by the section BA of the curve in which the surface pressure does not change while the adsorption increases from that corresponding to A to that corresponding to B. At B a second change occurs. For the portion of BC of the curve very great increases in surface pressure result from minute changes in adsorption. BC is accepted to represent a condensed film, AX a gaseous film, and AB a two-phase film consisting of islands of condensed film in an ocean of gaseous film. All these films are monomolecular in thickness with the molecules lying substantially flat in AX and increasingly erect in BC as the area per molecule is decreased.

If a similar situation existed in regard to the films of dodecylammonium ion on hematite it might well be expected that transformation at the equivalent of point B in Fig. 12 would be reflected in a break in the log Γ-log c curve, as drawn in Fig. 1. At this time it seems useful to call attention to this possibility, especially in the light of the ideas of two-dimensional mobility of adsorbate suggested by Hassialis and Myers.[19] Unfortunately the authors' measurements are still inadequate to justify pursuit of this intriguing suggestion.

On Contact Angle Measurements: Experimental data indicate that no simple relationship exists between adsorption density and contact angle θ. The apparent maximum value of θ, see Fig. 6, occurs at about the same adsorption density as the bends in the corresponding isotherms. Taggart et al[20] have attributed similar contact angle behavior to the adsorption of a second layer of collector by a mechanism involving the like-to-like attraction of hydrocarbon groups in the presence of a solvent.

The chief deterrent to a more exact correlation between contact angle and adsorption density stems from lack of information concerning the detailed distribution of the adsorbed agent in the solid-liquid interface. It seems reasonable to assume that collector ions held in the diffuse layer contribute relatively little to contact angle formation. On the other hand, the amount of reagent attached directly to the

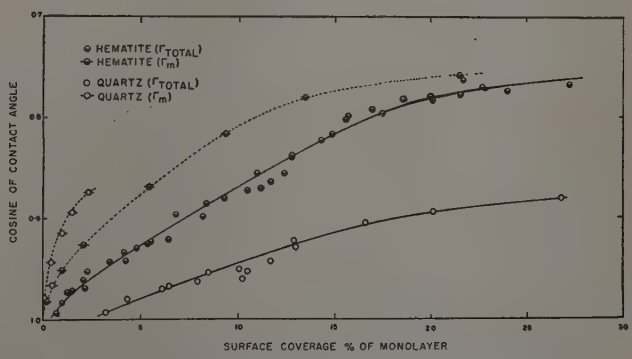


Fig. 13—Relationship between contact angle and adsorption coverage for hematite and quartz in dodecylammonium acetate solutions, both on the basis of total adsorption and molecular adsorption, Γ_m.

surface, aside from that in the double layer, has not yet been measured directly. Using adsorption Eq. 3, however, it is possible to estimate the value of Γ_m corresponding to any given value of Γ_{total}. Fig. 13 is a plot of cos θ vs Γ_{total} and cos θ vs Γ_m where Γ_{total} and Γ_m are expressed in percent of a monolayer. Contact angle data for the construction of this figure were obtained from Fig. 6.

Whereas it is quite clear that a definite adsorption density is required to obtain a finite contact angle in terms of Γ_{total}, the cos θ— Γ_m curves seem to pass through the origin in Fig. 13. This is what would be expected if in the absence of collector the adhesion between the liquid and solid were exactly equal to the cohesion of the liquid.

The existence of measurable contact angles and the attainment of excellent flotation with both hematite and quartz at total adsorbate surface coverages of only 4 to 5 pct of a monolayer (Γ_{total}) is of particular interest. Using Eq. 3 or Fig. 13 it appears that Γ_m for substantially complete flotation is well under 1 pct. Such a result is consonant with two ideas, 1—that the adsorbed entity is capable of lateral movement and 2—that it lies flat on the interface. Perhaps when contact is established the movable adsorbate moves so as to concentrate at the three-phase interface, and this may be why so incomplete a coating is nevertheless effective flotationwise. Such a situation would harmonize in part with W. O. Ostwald's[22] speculation of collector *adlineation* at a three-phase interline.

Summary

Using radio-assay technique and carbon-marked dodecylammonium acetate, an isotherm for the adsorption of amine from aqueous solution onto the surface of hematite has been established for a pH of 6 to 7 and a temperature of 21° to 24°C. At low concentrations log adsorption density is proportional to 0.6 log concentration. A bend occurs in the isotherm at a concentration of about 250 mg of amine salt per liter, corresponding to a surface coverage of 20 to 25 pct of a monolayer.

Reversibility of adsorption was established. As expected, adsorption density is independent of the specific surface of the mineral. The effect of pH on adsorption is small in acid solution, a ten thousandfold increase in hydrogen ion concentration decreasing adsorption about twofold.

Contact angles were measured on hematite and quartz surfaces immersed in aqueous amine acetate solutions using the captive bubble method. Temperature and pH were maintained essentially constant. The results are presented graphically and show the relation between contact angle θ and concentration and between cosine θ and surface coverage. No simple equation was found to connect adsorption with contact angle.

The work of adhesion for the mineral-air-liquid systems was calculated. Very flat maxima are found in the work of adhesion vs surface coverage curves at 0.35 and 1.05 parts of a monolayer for hematite and quartz respectively. The energy of adsorption is considerable for films very incompletely monomolecular, decreasing with increasing surface coverage actually changing in sign.

Acknowledgments

The authors wish to thank P. L. de Bruyn and D. W. Fuerstenau for their helpful suggestions and criticisms. They also express appreciation to the U. S. Atomic Energy Commission and to Armour & Co. for making this research possible.

References

[1] P. L. de Bruyn: Adsorption of Dodecylammonium Acetate on Quartz. Ph.D. thesis, Massachusetts Institute of Technology (May 1952).

[2] F. W. Bloecher, Jr.: A New Surface Measuring Tool for Mineral Engineers. MINING ENGINEERING, (1951) **3**, p. 255.

[3] C. S. Chang: Adsorption of Barium and Laurate Ions on Quartz. Ph.D. thesis, Massachusetts Institute of Technology (June 1951).

[4] H. J. Harwood and A. W. Ralston: The Synthesis of Lauric Acid and Dodecylamine Containing Carbon Fourteen. *Journal of Organic Chemistry* (September 1947) **12**, No. 5, pp. 740-741.

[5] S. C. Brown and W. W. Miller: Carbon Dioxide Filled Geiger-Muller Counters. *The Review of Scientific Instruments* (1946) **18**, No. 7, p. 496.

[6] A. M. Gaudin, P. L. de Bruyn, F. W. Bloecher, Jr., and C. S. Chang: Radioactive Tracers in Flotation. *Mining and Metallurgy* (August 1948) **29**, p. 432.

[7] F. W. Bloecher, Jr.: Concerning the Adsorption of Dodecylamine on Quartz. M.A. thesis, Massachusetts Institute of Technology (June 1949).

[8] A. F. Taggart, T. C. Taylor, and C. R. Ince: Experiments with Flotation Reagents. *Trans.* AIME (1930) **87**, p. 285-368.

[9] Guido R. M. delGuidice: The Bubble Machine for Flotation Testing. *Engineering and Mining Journal* (1936) **137**, p. 291.

[10] R. Schuhmann, Jr., and B. Prakash: Effect of $BaCl_2$ and Other Activators on Soap Flotation of Quartz. *Trans.* AIME (1950) **187**, p. 591.

[11] Robert B. Dean: The Gibbs—A Rational Unit for Adsorption. *Journal of Physics and Colloid Chemistry* (1951) **55**, pp. 611-612.

[12] Linus Pauling and Sterling B. Hendricks: The Crystal Structure of Hematite and Corundum. *Journal of the American Chemical Society* (1925) **49**, pp. 781-790.

[13] Ralph W. G. Wyckoff: *Crystal Chemistry*. Vol. I, Chap. V, text p. 11, illus. p. 3. Interscience Publishers Inc., New York, 1951.

[14] Herbert H. Kellogg, and Hugo Vasquez-Rosas: Amine Flotation of Sphalerite-Galena Ores. AIME (1946) *TP 1906*.

[15] Everett J. Hoffman, G. E. Boyd, and A. W. Ralston: Studies on High Molecular Weight Aliphatic Amines and Their Salts. VIII. Soluble and Insoluble Films of the Amine Acetates. A. The Surface Tension of Aqueous Solutions of Dodecylammonium Acetate, *Journal of the American Chemical Society* (1940) **62**, p. 2375.

[16] A. W. Ralston, C. W. Hoer, and C. Hoffman: Studies on High Molecular Weight Aliphatic Amines and Their Salts. IV. Electrical Conductivities of Aqueous Solutions of the Hydrochlorides and Acetates of Dodecyl— and Octadecylamines. *Journal of the American Chemical Society* (1940) **62**, pp. 97-103.

[17] N. K. Adam: *The Physics and Chemistry of Surfaces*, p. 50. Oxford University Press, London, 1941.

[18] E. J. W. Verwey, and J. Th. G. Overbeek: *Theory of the Stability of Lyophobic Colloids*. Elsevier Publishing Co. Inc., 1948.

[19] M. D. Hassialas, and C. G. Meyers: Collector Mobility and Bubble Contact, *Trans.* AIME (1951) **188**, pp. 961-968.

[20] A. F. Taggart, N. Arbiter, and H. H. Kellogg: The Mechanism of Collection of Metals and Metallic Sulphides by Amine Salts. *Trans.* AIME (1943) **153**, p. 517.

[21] A. M. Gaudin, and F. W. Bloecher, Jr.: Concerning the Adsorption of Dodecylamine on Quartz. *Trans.* AIME (1950) **187**, pp. 449-505.

[22] W. O. Ostwald: Theory of Flotation. *Kolloid Z.* (1932) **58**, pp. 179-183; **60**, pp. 324-340.

[23] John G. Morrow: Adsorption of Dodecylammonium Acetate on Hematite and Sphalerite. Ph.D. thesis, Massachusetts Institute of Technology, 1952.

Magnetic Attraction of Stacked Drill Rods

by John L. Baum

Tests show that 50 times the earth's normal field can exist near stacked drill rods. Protection against the effect of these strong fields can be obtained by means of a removable sleeve of common iron pipe.

GEOLOGISTS and engineers working around a diamond drill rig have often had the opportunity to observe the magnetic attraction of drill rods pulled out of the hole for core removal. This is sometimes an amusing pastime, but this same magnetic force inadvertently applied to a compass needle can cause it to lose its polarity entirely or to become reverse polarized when placed in the immediate vicinity of stacked rods. The first recommendation, of course, would be to avoid the rods, but in surveying an operating drillhole this is not always possible; therefore steps must be taken to neutralize the magnetic effect of the drill rods.

Early in the summer of 1949 the geological department of the New Jersey Zinc Co. undertook at Franklin, N. J., an extensive program of surveying the course of deep diamond drillholes. The majority of the holes were surveyed while in the process of being drilled, and considerable difficulty was encountered. A pattern for the deviation of drillholes had been established for the district from surveys in holes from which the drill rig had been removed. It was observed that a certain operating deep drillhole being surveyed did not fit this pattern. In the resulting confusion of repeated surveys and cementing a plug for wedging operations, the lower 1800 ft of the hole was lost. Because of this incident studies were initiated to determine the source of the difficulties and to develop safeguards against recurrence of faulty surveys.

J. L. BAUM is Chief Geologist, Franklin-Sterling Operations, the New Jersey Zinc Co., Franklin, N. J.

Discussion on this paper, TP 3953I, may be sent (2 copies) to AIME before Feb. 28, 1955. Manuscript, Oct. 13, 1953.

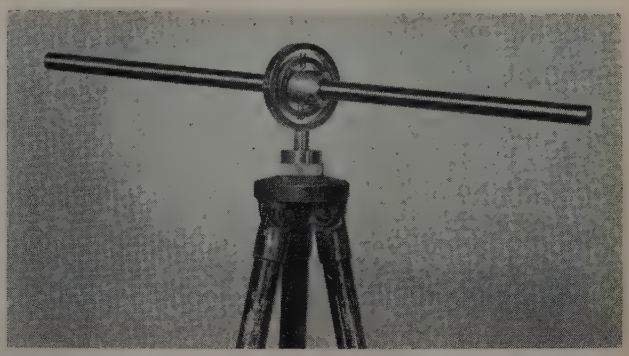
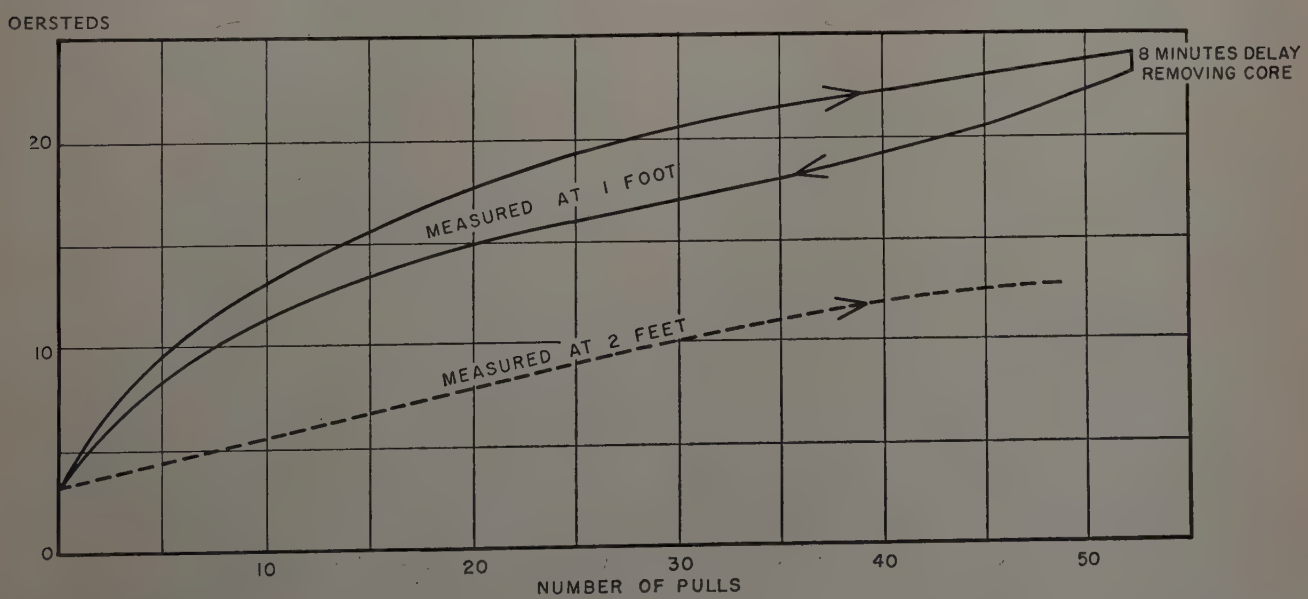


Fig. 1—Oscillating magnet mounted for field measurements.

Using a hand magnet easily shows that the polarity of a compass needle can be reversed if rotation of the needle is hindered through tilting of the compass or clamping of the mechanism. Such experiments are not recommended, especially in the case of the clockwork type of instruments. A magnetized hairspring in a clockwork instrument will cause the compass card to assume a periodic motion which will not stop until the clockwork runs down.

Routine check readings of instrument north before and after each survey are not always sufficient to catch instances of reversed polarity of the compass needle. Although the majority of instances probably take place while the instrument is being removed from the hole with needle in fixed position, it is demonstrable that a sluggish needle, weakened during a previous test, can be reverse polarized

Fig. 2—A drill rods, 30-ft pulls.



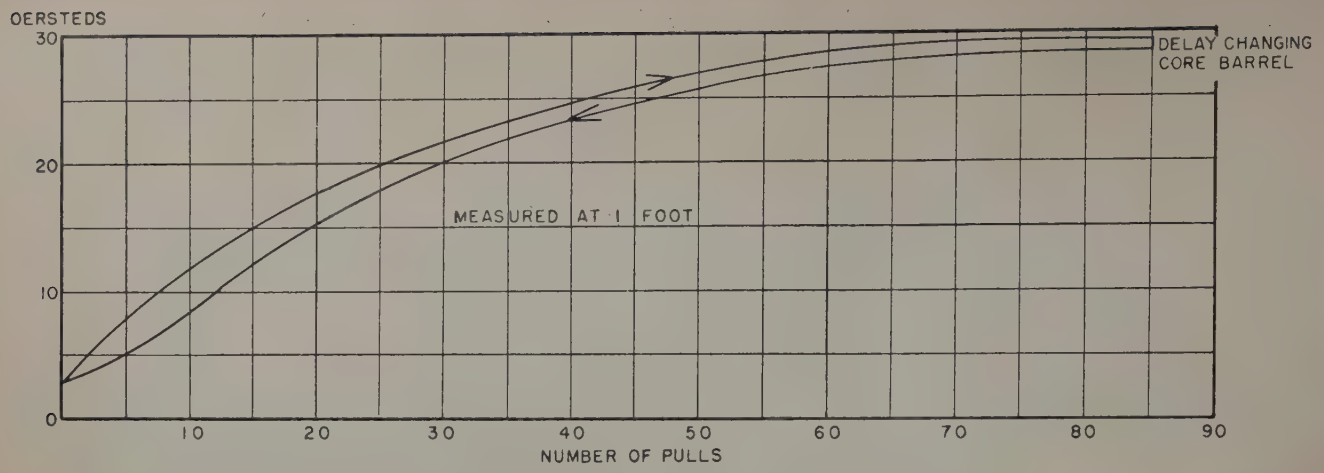


Fig. 3—*B* drill rods, 60-ft pulls.

while being placed into position for lowering. In the case of polarity reversed both before and after a test, the introduced error defies detection by any means other than repeated surveys.

Assistance of the New Jersey Zinc Co. research department at Palmerton, Pa., was requested, and R. K. Waring of the research staff kindly offered a number of suggestions. He stated that manufacturers of drillhole survey equipment can make a contribution to partial solution of the problem by incorporating a magnetic needle of greater permanence and higher strength than was furnished in the devices tested. Waring also pointed out that experiments show that surrounding a compass with soft iron, such as a piece of pipe, will shield it from the effect of a magnetic field.

At the drill rig a length of pipe split longitudinally and hinged for easy removal is now used. This magnetic shield hangs on the instrument container by virtue of a constriction welded on top of the pipe. When the container enters the drillhole casing projections hold back the shield. It can then be removed if desired, to be replaced when the instrument container is withdrawn. Loading of the container is accomplished 5 ft or more from the stacked rods. Because the effect of a magnetic casing has not caused trouble in surveys where the field of the stacked rods has been neutralized, little effect is ascribed to the casing in influencing the accuracy of survey readings.

Instrument to Measure Field Strength

The research department was requested to develop a method of measuring the actual field strength of magnetic forces which act upon survey compasses. The oscillating magnet was selected for this purpose because of simple construction and operation. The period of oscillation depends on the magnetic moment of the magnet and the strength of the field in which it oscillates. The first magnet tested at Franklin worked well, checked against later results, but the device was hung only on a thin nichrome wire, which failed frequently.

The second instrument, Fig. 1, consists of a rod of 36 pct cobalt steel, ⅜ in. diam and 30 cm long, mounted in a cylinder. The cylinder is pivoted at top and bottom within an inner gimbals ring, which in turn is supported in an outer rigid gimbals ring. Miniature ball bearings are used.

The cobalt-steel bar was heat-treated by standard procedures and then magnetized. The device was calibrated in a magnetic field of known strength.

Field Strength Measured

A series of measurements were taken at several operating drillholes with this calibrated instrument to determine the field strength encountered near the rods. Precautions were taken to obtain accurate data and to avoid any outside influences.

From the data accumulated in field measurements it is apparent that a number of variables affect the recorded magnetic field of stacked drill rods. In order of decreasing importance these are: distance of the instrument from the rods, number of rod pulls, and length of time the rods have been out of the hole. Of minor importance are the size of the rods, the length of each pull, and the manner in which the stacked rods are grouped. Graphs prepared from some of the data illustrate a few of the possible modifications of values that can be ascribed to one or more of the above listed variables, see Figs. 2 and 3.

Measurements show that stacked rods at a distance of 1 ft can exert a force of almost 30 oersteds, or 50 times the earth's normal field. At 2 ft, other conditions being equal, the force is nearly 18 oersteds. Values at 3 ft can be 6½ oersteds, and at greater distances the force is relatively slight. Stacked rods lost part of their magnetism quickly, and during the period of core removal were observed to lose up to 1¾ oersteds. As might be expected, the bottom of a stack of drill rods is the north pole of the bar magnet produced.

Examination of the accompanying illustrations suggests that a magnetic field of a value in excess of 30 oersteds is not likely to be encountered a foot or more from any stack of diamond drill rods used in current practice. This is because increase in size of the magnetic source is ultimately offset by loss of polarity during the length of time it takes to hoist a large number of rods. It follows, therefore, that 30 oersteds probably represent a maximum value for magnetic field strengths at a distance of 1 ft under present hoisting speeds.

Acknowledgment

The author wishes to express his appreciation to the New Jersey Zinc Co. for permission to publish this article and to the company's research department for their technical assistance.

Near-Surface Hydrocarbons and Petroleum Accumulation at Depth

by Leo Horvitz

Microanalysis of near-surface soils for hydrocarbons is the basis of a method for locating gas and oil deposits. To substantiate this technique, evidence of vertical migration of hydrocarbons from petroleum accumulations is presented. Tabulated data relevant to hydrocarbon surveys conducted in several petroleum provinces are included.

PETROLEUM and natural gas are composed principally of the saturated hydrocarbons ranging from methane, the lightest, to nonvolatile liquids and solids containing approximately thirty-five carbon atoms. A technique for locating buried accumulations of these hydrocarbons before drilling obviously requires that some of the hydrocarbons leave the deposit and migrate toward the surface of the earth where they may be detected in their original form.

Earliest attempts to link near surface hydrocarbons to petroleum at depth were apparently made by Laubmeyer[1] in Germany and by Sokolov[2] in Russia. These investigators collected samples of soil air from boreholes one to two meters deep and analyzed them for traces of hydrocarbons. They found that soil air over producing areas is richer in these constituents than is soil air over barren areas.

Since 1936 work on petroleum exploration techniques of this type has been going on in this country. However, instead of determining hydrocarbon content of soil air collected in the field, investigators analyze samples of the soil itself[3-6] for adsorbed and occluded hydrocarbons, which are released by suitable treatment and found in larger amounts than are the quantities reported for soil air. Difficulties often encountered in collecting gas samples in the field, moreover, are eliminated when soil is used as the sampling medium.

Field Procedure: Sample locations are first surveyed over the area to be investigated. Care is taken to locate the stations at considerable distances from roads, pipelines, drilling wells, and other sources of contamination. The borehole may be dug with a bucket-type hand auger or with mechanical drilling equipment. Lubricants are avoided in either case. When the desired depth is reached, a sample is brought to the surface, placed in a pint glass jar or can, and securely sealed. Sample containers are carefully labeled and delivered to the analytical laboratory. Generally a satisfactory sampling depth range is 8 to 12 ft. In some regions, however, satisfactory data are obtained from samples collected at much shallower depths. Such is the case, for example, in areas of west Texas where the limestone and caliche near the surface occlude hydrocarbons and prevent their rapid escape to the atmosphere.

In carrying out broad reconnaissance surveys in search of large features, considerable time is saved by first taking samples one-fourth to one-half mile apart along profiles about one mile apart. If the analytical data indicate a hydrocarbon anomaly of interest, additional samples are taken to produce a more dense and uniform sampling pattern within the interesting area. This sampling program is particularly adaptable to areas that are sectionized. In areas covered with a network of roads, sampling along these roads facilitates the reconnaissance survey. Actual sampling density used depends upon areal extent of features expected. When flanks of piercement-type domes where accumulations may be only several hundred feet wide are sampled, stations are often no more than 200 ft apart.

Analytical Technique: Of the hydrocarbons composing petroleum, only the more volatile would be expected to reach the surface of the earth. The analytical technique, therefore, was developed to determine only those constituents that exert a vapor pressure at room temperature. Actually, in near-surface soils, only a very small part of the hydrocarbons are heavier than pentane. Details of the analytical technique have previously been reported.[6, 7] Only a brief description of the methods will be presented here.

A weighed portion of the sample, about 100 g, is first treated with an aqueous solution of copper sulphate and then with phosphoric acid in a partial vacuum. The copper sulphate prevents the reaction of the acid with carbides that may be present because the sample has been contaminated by auger particles. Such a reaction may produce spurious methane. The role of the acid is to decompose any carbonates present, thereby helping to release the hydrocarbons. The carbon dioxide is removed with potassium hydroxide and the flask containing the

L. HORVITZ, Member AIME, is Consulting Chemist and Geochemist, Horvitz Research Laboratories, Houston, Tex.

Discussion on this paper, TP 3927L, may be sent (2 copies) to AIME before Jan. 31, 1955. Manuscript, Feb. 9, 1954. New York Meeting, February 1954.

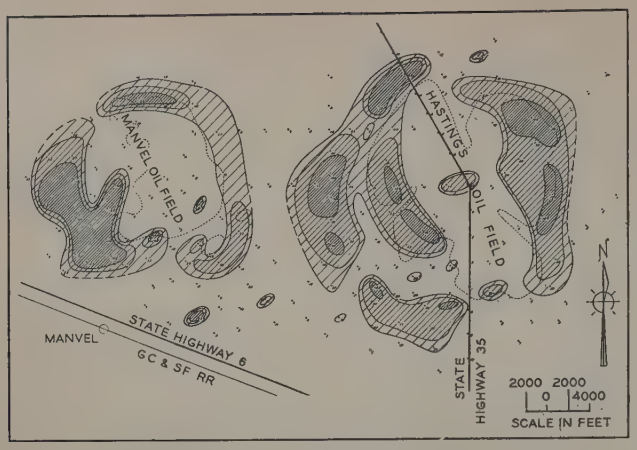


Fig. 1—Hydrocarbon survey of the Manvel and Hastings oil fields, Brazoria County, Texas.

sample is then heated 30 min. at 100°C. This mild heat treatment does not permit decomposition of organic matter that may be present. The carbon dioxide free sample is collected in a previously evacuated tube and analyzed.

To analyze the extracted gases, low temperature fractionation and combustion methods are employed. The gaseous mixture is passed through potassium hydroxide solution, concentrated sulphuric acid, ascarite, and phosphoric anhydride before entering the analytical apparatus proper. The concentrated sulphuric acid removes the oxygen-containing compounds referred to as *pseudo-hexane* in a previous paper.[4] The routine analysis has been standardized to include two separate fractions, 1—methane and 2—ethane and heavier hydrocarbons. The methane is determined separately because this hydrocarbon may be present in the soil as a result of decaying organic matter. In conjunction with appropriate traps and valves, the separation is conducted at the temperature of liquid nitrogen, —196°C. Methane is gaseous at this temperature, whereas ethane and heavier hydrocarbons are condensed. The methane is determined by combustion of the noncondensable mixture over a glowing platinum wire. The resulting carbon dioxide is collected and measured and from its quantity the methane is readily computed. The quantity of ethane and heavier hydrocarbons is determined by first vaporizing and measuring the volume of the condensed fraction and then measuring the carbon dioxide resulting from combustion of this fraction with purified air. The increase in the final volume is a measure of the quantity of ethane and heavier hydrocarbons initially present. Sensitive McLeod gages are employed to measure the pressures from which the volumes of hydrocarbons are calculated.

The total volume of the gas sample initially prepared is known and the various parts of the analytical apparatus are calibrated, permitting calculation of volumes of gas used for the different determinations. Volumes of the various constituents are thus readily determined. Weights of the different fractions are calculated from the gas laws and the final results expressed in parts per billion by weight (dry basis) of the soil sample. Data are expressed in these units for convenience. They may be expressed in other terms, for example, in cubic centimeters per kilogram. For the case of ethane and heavier hydrocarbons (expressed as butane) one part per billion by weight is equivalent to approximately 0.0004 cc per kilogram of soil.

Analyses of duplicate portions of numerous soil samples yield values for the duplicates that agree within limits of 20 pct for the range of 10 parts per billion and above. For the case of very low values, the percentage differences are of course much wider.

Hydrocarbon Distribution Patterns: Data obtained by the soil analysis technique indicate the usual hydrocarbon distribution pattern over a large producing area to be one in which low concentrations are found over the center of the productive area, whereas relatively high concentrations occur at the edges. Outside the bands of high concentration, low values are again obtained. Occasionally an anomaly surrounded by a background of low values is found in which high values are associated with the entire productive area. This type is usually associated with accumulations of relatively small areal extent. Another pattern sometimes encountered is one in which values found over the productive area are considerably higher than those obtained in the background areas, with still higher concentrations at the edges of the producing area.

Only a weak anomaly, and often none, is found over a depleted field. Apparently when the source at depth begins to disappear, hydrocarbons near the surface also begin to disappear.

Fig. 1 shows the results of an experimental survey conducted in 1946 over the Manvel and Hastings oil fields, Brazoria County, Texas. Samples were collected at depths ranging from 8 to 12 ft. Ethane and heavier hydrocarbon values, expressed in parts per billion by weight, have been plotted at the station location symbols. The following groups of values are enclosed by the various contour lines: 25 to 49; 50 to 99; 100 and above. To display the variations in hydrocarbon concentration over the area sampled several degrees of shading have been used. Values below 25 parts per billion by weight are in the unshaded areas, values from 25 to 49 parts per billion in the areas of lightest shading, values from 50 to 99 in the zones of intermediate shading, and values of 100 parts per billion and above in the most heavily shaded areas. The broken lines, within the anomalous areas, represent the outer limits of the producing areas of the two fields.

The hydrocarbon anomalies are distinct and of the more general type. The anomaly associated with the Hastings field is considerably stronger than that over the Manvel field. The Hastings field, discovered in 1934, is one of the largest in Texas and only a relatively small part of its reserve has been produced. The Manvel field, discovered in 1931, has a much smaller reserve and has produced for a longer time. The low values over the main producing areas suggest that no contamination of the soil was caused by the producing wells.

The anomaly associated with the Hastings field contains a double band of high hydrocarbon concentration on the west side. The inner band is apparently associated with the west side of the present producing area, while the outer band, which extends beyond the producing area, may be associated with a new, potentially productive section. On the west side of the Manvel field a possible extension is also indicated. The main producing zones of the Manvel and Hastings fields are in the Frio formation. The anomalous conditions on the west flanks of these fields may reflect accumulations at greater depth, perhaps in the Vicksburg or Yegua formations.

This survey indicates the importance of the acid treatment in the extraction procedure. In an earlier

survey[4] of the Hastings field conducted in 1937, prior to the introduction of this improvement, values of a much lower magnitude were obtained, and although an anomaly developed a much less distinct pattern resulted. Apparently heating of samples without the acid treatment liberates only the loosely held hydrocarbons.

It is of interest to point out that the methane data yielded patterns over the Manvel and Hastings fields similar to those obtained from the ethane and heavier hydrocarbons. Methane is not considered significant in marshy areas but is sometimes the most significant constituent determined in near-surface soils. From a dry gas deposit, for example, only methane emanates in significant amounts and produces the near-surface anomaly.

Fig. 2 shows a portion of a very large survey conducted in Harris, Brazoria, and Galveston Counties, Texas, and completed in 1952. The portion of the survey which is presented includes 227 stations which were sampled at depths of 8 to 12 ft and covers approximately 25,000 acres. This survey illustrates the type of data obtained and the interpretation made prior to the discovery of petroleum accumulation. Values shown on the map are for the ethane and heavier hydrocarbons, expressed in parts per billion by weight. The same contour levels and shading system were used as for the area in Fig. 1.

Four hydrocarbon anomalies, A-1, A-2, A-3, A-4, and three leads, L-1, L-2, L-3, are indicated on the map. This survey is particularly interesting because it contains anomalies of the different types that have been encountered. Anomaly A-1 is of the type containing an area of low concentrations bordered by high values; A-2 is suggestive of the type made up of an area of hydrocarbon concentration greater than the background, with still higher concentrations at the edges of the anomaly; A-3 is made up of an area of high concentrations; and anomaly A-4 appears to be a combination of two types, the northern half being similar to A-1 and the southern part similar to A-3. To define the leads, more data are obviously needed. For example, for the case of lead L-2, data are required within the lake area, outlined on the map, and in the areas immediately to the north and west of the lead. Collection of samples within the lake area was not attempted and permission to sample outside the area of the lead could not be obtained at the time the survey was conducted.

The nonproductive wells drilled prior to the completion of the survey were found to be located in the background. Of the five wells drilled after completion of the survey, only one was located within a hydrocarbon anomaly. This was the No. 1 Hecker well, which was completed as a gas distillate discovery. The commercial value of this new accumulation will not be known, of course, until additional development occurs.

From the data of Fig. 2 the average background value is calculated to be only eight parts per billion while the average anomalous value is 145 for A-1, 74 for A-2, 101 for A-3, and 81 for A-4. Ratios of the anomalous values to the background, therefore, range from 9 to 18. Another interesting observation is that the first contour level, containing values from 25 to 49 parts per billion by weight, may be omitted without producing significant variations in the anomalies.

The average background value is not constant for all areas and can be determined to a reasonably accurate degree only if a large area is sampled. The determination of significant anomalous values for a particular area may be facilitated by extending the survey, where possible, to include part of a productive area. Generally the background values for areas in the Gulf Coast are low, usually below 25 parts per billion. In many sections of north Texas and west Texas, and other petroleum-bearing regions, however, average background values may approach 100 parts per billion for ethane and heavier hydrocarbons. In such areas of high background concentration, the anomalous areas are still readily recognized if sufficient data are available. The average of the values that are usually associated with a significant anomaly is of the order of 300 pct, or more, greater than the average background value.

Vertical Migration of Hydrocarbons from a Petroleum Accumulation: A direct relationship between the data presented in Figs. 1 and 2 and petroleum accumulation at depth would be admitted readily if hydrocarbon leakage from the accumulation to the surface of the earth could be demonstrated. An obvious method of determining whether or not such migration occurs involves the collection and analysis of formation cores from the surface to the petroleum accumulation and the comparison of the hydrocarbon data obtained from these cores with similar data for cores taken from wells drilled in barren areas. Unfortunately, continuous cores of the type required are never available; usually cores are available for only very short sections of wells. Formation cuttings are, however, always produced during the drilling of wells. A disadvantage of this sample medium is that the cuttings are washed with hot mud as they are pumped to the surface, which causes an appreciable reduction of their hydrocar-

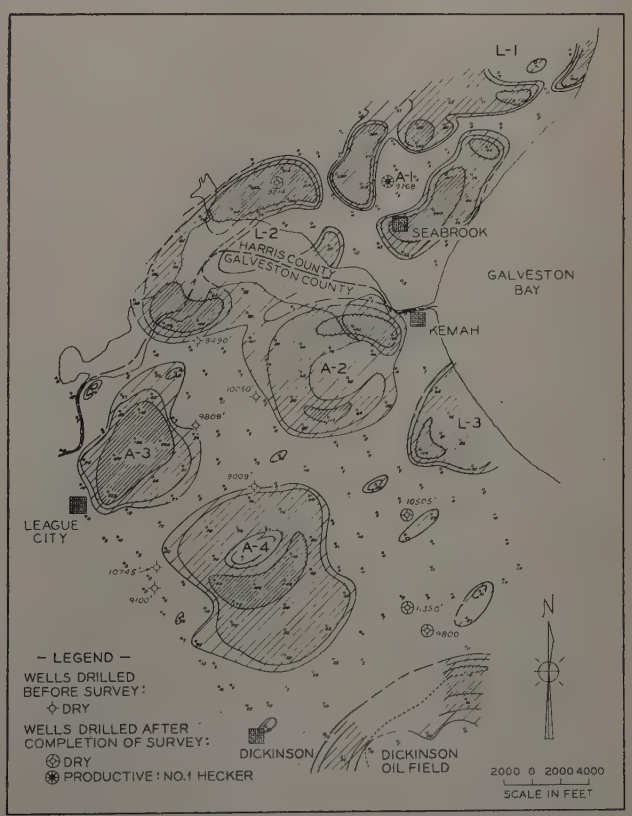


Fig. 2—Survey of an area in Harris and Galveston Counties, Texas, which illustrates the various types of hydrocarbon distribution patterns encountered. Encircled wells were drilled after completion of survey.

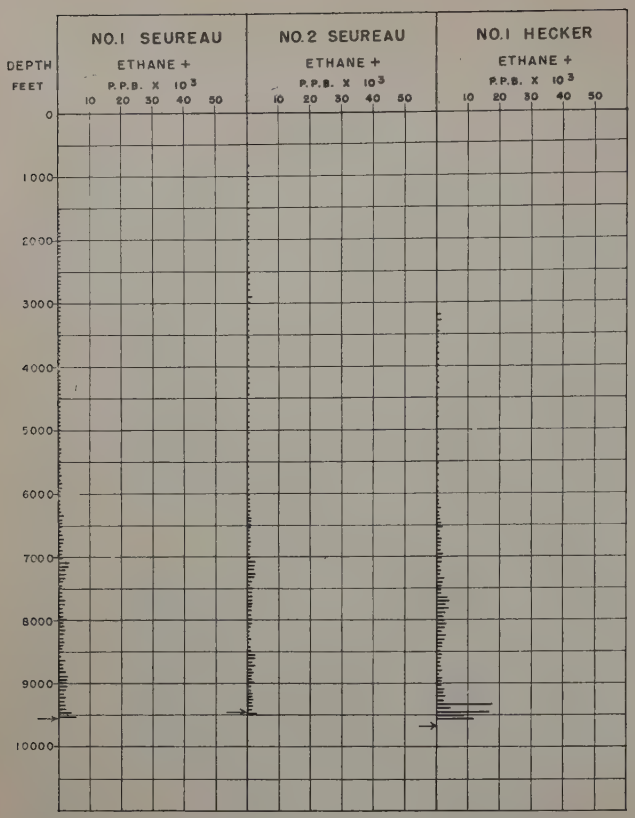


Fig. 3—Hydrocarbon logs of three wells drilled in the Seabrook area, Harris County, Texas. The Hecker well encountered a gas condensate zone at 9690 ft, indicated by arrow. Barren equivalent zones in other two wells are also indicated by arrows. Only apparent source for high hydrocarbon values in lower part of Hecker log is petroleum accumulation below.

bon contents. In addition, data for the upper 2000 to 3000 ft of section drilled are not usually available because this section, especially along the Gulf Coast, is composed of soft clays and sands and returns from the well are thoroughly mixed with the drilling mud and impossible to separate. In deeper portions of the well firm shale and consolidated sands are encountered which can be readily separated from drilling fluid. In spite of these objectionable characteristics, a great many samples from many different wells and formations have been collected and significant data have been obtained from them.

Normally samples are collected from the shale shaker at the end of each 30 ft of section drilled. The samples are placed in pint glass jars or sealed in cans and brought to the laboratory where they are washed free of drilling fluid. Composite samples are usually prepared for each 60 to 90 ft of drilling. When more detailed information is required a greater sampling density is used.

The gas extraction and hydrocarbon analysis procedures used in connection with the cuttings are the same as those described for the determination of hydrocarbons in near surface soils. Just as in that case, undried samples are used and the moisture content, needed in the calculations, is determined from a portion of the sample separate from that used in the analysis. Usually the methane, the ethane and heavier hydrocarbon data, and the total hydrocarbon data are plotted in the form of a log. A scale of 1 in. to 1000 ft is used so overall patterns that develop can be viewed. Additional information can be obtained by separating the ethane and heavier hydrocarbons into ethane-propane-butane and pentane and heavier hydrocarbon fractions.

In Fig. 3 are shown the plotted ethane and heavier hydrocarbon data obtained from cuttings collected from three wells drilled in the Seabrook area of Harris County, Texas. The No. 1 Seureau is located about 2800 ft north of the No. 2 Seureau, and the No. 1 Hecker well, which appears on Fig. 2, is located approximately two miles from the No. 2 Seureau in a southwesterly direction. The two Seureau logs are generally similar. The log of the Hecker well is different in that much greater hydrocarbon values were obtained for the bottom several hundred feet of this well. The only apparent explanation for this difference is the fact that a gas distillate accumulation, noted on Fig. 3 by an arrow, was encountered at 9690 ft in the Hecker well, whereas the corresponding zones in the other two wells were nonproductive. The zones in the Seureau wells that correspond to the Hecker zone are also indicated by arrows and were determined by correlation of the electrical logs of the three wells. Cuttings for the section immediately above the Hecker accumulation were not taken. When the three logs down to the Hecker zone equivalent are considered, it is apparent that a petroleum reservoir produces a measurable effect for a considerable distance above the deposit. If the hydrocarbons in the lower part of the Hecker well were indigenous, then the same order of magnitude of values should have been obtained in the corresponding sections of the other two wells.

Table I. Summary of Exploration Experience

Area	Coverage in Acres	Anomalies over Producing Fields Covered by Surveys: Present	Anomalies over Producing Fields Covered by Surveys: Absent	New Anomalies Revealed by Surveys	New Anomalies: Drilled	Results: Productive	Results: Dry
Texas Gulf Coast	1,018,000	19	0	45	20	13	7
North Texas	201,000	6	2	8	4	3	1
South Texas	64,000	4	0	5	4	2	2
West Texas	283,000	4	0	9	3	2	1
Louisiana	219,000	1	0	11	4	2	2
Mississippi	239,000	2	0	5	2	1	1
Georgia	750,000	0	0	10	0	0	0
Florida	50,000	0	0	1	1	0	1
California	30,000	1	0	5	0	0	0
South America: Ecuador	380,000	0	0	10	1	0	1
Canada: British Columbia	90,000	Large anomalous area with bands of high concentration parallel to strike. Scattered gas wells associated with areas of low concentration between these bands. Hydrocarbon survey has been confirmed by additional development.					
Totals	3,324,000	37	2	109	39	23	16

An interesting set of logs is shown in Fig. 4. Data were obtained from cuttings collected from three wells drilled in the North Thompson field of Fort Bend County, Texas. Unfortunately, only samples from the bottom 450 ft of the 1-B Scanlan well were available for analysis; nevertheless, a distinct buildup in hydrocarbon concentration is apparent above the oil reservoir, which was encountered at 7957 ft. This well was completed in the Vicksburg formation from perforations at 7957 to 7965 ft as an excellent producer. The 1-C and 1-E wells, located 2600 ft northeast and 2100 ft east of the 1-B, respec-

tively, and outside the producing area, encountered salt water with only small amounts of oil in this zone. The values, in the sections immediately above the producing horizon equivalent, are considerably lower than those in the corresponding section of well 1-B. These data again serve to suggest that hydrocarbons migrate vertically in significant quantities from a petroleum reservoir.

Many logs similar to those shown in Figs. 3 and 4 have been prepared and some of them appear elsewhere.[8, 9] While the nature of the sampling medium makes it impossible to follow the hydrocarbons to the surface, cases have been observed in which appreciable values have been obtained for several thousand feet above the accumulation. Wells located at the edges of petroleum deposits often produce longer sections of relatively high hydrocarbons than do wells that are centrally located.

Exploration Experience: Because of the difficulties encountered in definitely establishing either a theoretical or experimental basis for vertical leakage of hydrocarbons from a petroleum accumulation to the surface, the value of soil hydrocarbon data in exploration must be determined experimentally by the accumulation and study of a great deal of data. During the 11-year period ending in 1953 a total of 130 hydrocarbon surveys have been conducted. Of these, 61 covered 20,000 acres or more. In Table I only data relevant to the group of larger surveys are included because an area of at least 20,000 acres should be sampled to permit an adequate interpretation. The status of the areas at the time the surveys were completed is noted and new development that has taken place since is indicated.

The table shows that of 39 anomalies considered to be associated with oil or gas accumulation, 59 pct were confirmed. Of the 69 smaller surveys conducted, a number revealed significant anomalies, and of 11 anomalies drilled, 4, or 36 pct were found to be productive.

While discoveries of new oil or gas deposits made on the basis of hydrocarbon surveys alone are included in Table I, credit for all the discoveries is not claimed. For purposes of evaluation, however, the technique was credited with a confirmation if a petroleum accumulation was found within an anomaly provided that the survey was completed prior to drilling. Similarly, a failure was recorded when a nonproductive well was drilled within a hydrocarbon anomaly.

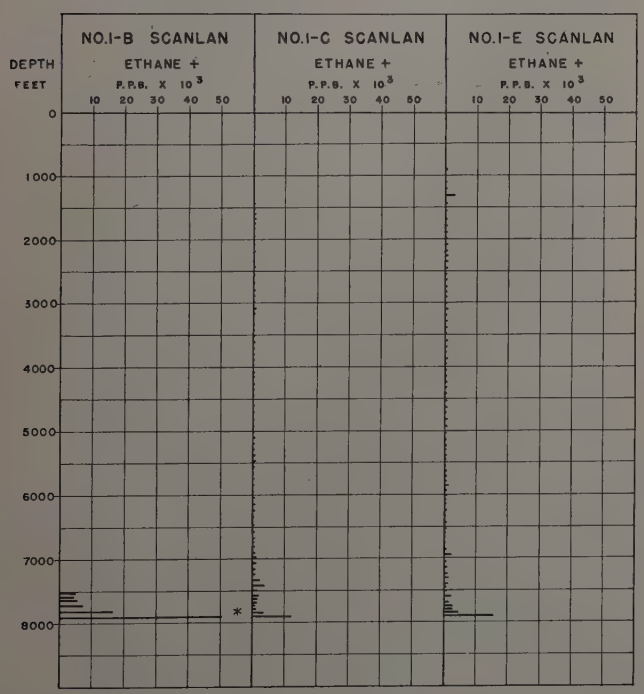


Fig. 4—Hydrocarbon logs of three wells located in the North Thompson field, Fort Bend County, Texas. The No. 1-B Scanlan is the only producer and shows much greater hydrocarbon values in the section above the productive horizon than do the others. Vertical migration of hydrocarbons from a petroleum accumulation is strongly suggested by the 1-B log. Lowest line, marked by asterisk, represents off-scale value of 72,640 parts per billion by weight.

Conclusions

The technique described in this paper differs from all other methods of exploration in that it involves measurement of some of the very constituents that make up natural gas and oil deposits, namely, the saturated hydrocarbons. Data presented offer evidence that the hydrocarbons observed near surface originate in buried deposits, suggesting a direct approach to locating petroleum.

Because no single method of exploration is believed to be universally applicable, this technique should be used in conjunction with others, particularly with subsurface geology and the seismic method. Hydrocarbon surveys are particularly important in the reconnaissance phase of exploration whereby anomalies which may serve as targets for the seismic method may be located. While hydrocarbon data may serve to outline a potential oil or gas accumulation, no evidence as to the depth to the accumulation and no structural information can be obtained from these data. Seismic data can throw light on these problems, but in the event a strong hydrocarbon anomaly is found not to be associated with structure, the possibility that it is reflecting a stratigraphic trap should not be overlooked.

Acknowledgment

The writer wishes to express his thanks to Beersheba Corp. for use of the data shown in Fig. 3 and to Christensen and Matthews for release of the hydrocarbon logs of the North Thompson field.

References

[1] G. Laubmeyer: A New Geophysical Prospecting Method, Especially for Deposits of Hydrocarbons. *Petroleum* (1933) **29**, pp. 1-4.

[2] V. A. Sokolov: Summary of the Experimental Work of the Gas Survey. *Neftyanoye Khozyaystvo* (1935) **27**, pp. 28-34.

[3] E. E. Rosaire: Shallow Stratigraphic Variations over Gulf Coast Structures. *Geophysics* (March 1938) **3**, pp. 108-109.

[4] Leo Horvitz: On Geochemical Prospecting. *Geophysics* (July 1939) **4**, pp. 210-225.

[5] Leo Horvitz: Recent Developments in Geochemical Prospecting for Petroleum. *Geophysics* (October 1945) **10**, pp. 487-493.

[6] Leo Horvitz: Chemistry in Exploration for Petroleum. Preprint in *Papers presented before the Petroleum Division*, American Chemical Society, St. Louis Meeting, April 7-11, 1941.

[7] J. J. Jakosky: *Exploration Geophysics*, 2nd ed., pp. 944-948. Trija Publishing Co., 1950.

[8] Leo Horvitz: Geochemical Well Logging. *A Symposium of Subsurface Logging Techniques*, pp. 89-94. University of Oklahoma, 1950.

[9] E. E. Rosaire: Geochemical Prospecting for Petroleum. Bulletin of the *American Association of Petroleum Geologists* (August 1940) **24**, pp. 1418-1426.

Variable resistivity in a surface layer may lead to misinterpretation. Errors can sometimes be avoided if the effect can be recognized and accounted for.

Effect of a Variable Surface Layer on Apparent Resistivity Data

by Harold M. Mooney

WHEN apparent resistivity data are taken with the symmetrical Wenner 4-electrode spread, a fixed center position is used and readings are taken for values of electrode separation. Basic data consist of apparent resistivity plotted against separation of adjacent electrodes . The interpreter attempts to infer geologic structure, such as the depth to discontinuities and the nature of subsurface earth materials.

An earlier paper[1] described methods for interpreting resistivity data. All of these involve a severe assumption, namely that the earth in the region of interest consists of horizontal layers, electrically homogeneous and isotropic. The actual earth never satisfies this assumption exactly and may deviate from it so much that none of the above methods can be applied. Attempts in three directions have been made to modify the assumption so that it approaches known geologic complexity more closely.

First, curves have been calculated for dipping discontinuities. Stern[2] and Aldredge[3] chose a few widely separated dip angles. Trudu[4] confined his attention to small dips. Berel'kovskiy and Zubanov[5] computed gradient curves for widely separated dip angles. Unz[6] has given the most complete solution, with a brief attempt to treat the three-layer case.

Second, anisotropy can be taken into account. It seems geologically probable that layered materials have different vertical and horizontal conductivities. Cagniard,[7] Maillet,[8] and Pirson[9] set up methods for finding an equivalent hypothetical isotropic medium. Standard interpretation methods can be applied to this, and the actual medium can then be deduced. Belluigi[10] discounts the practical importance of anisotropy; Geneslay and Rouget[11] do not agree with his conclusion.

Third, the effect of variable resistivity in a layer can be considered. Keck and Colby[12] examine the mathematics of an exponential increase in a surface layer. Several authors, for example, Stevenson,[13] consider a continuous variation of resistivity with depth. The present paper deals with a linear variation of resistivity in a surface layer.

Geologically, surface variations should be expected. Unconsolidated materials such as glacial drift show marked irregularities over short distances. The effects of weathering change with depth. The moisture content of material above the water table may vary from a dry sand to a saturated clay, and both of these will be changed by rainfall.

Figs. 1 to 6 present apparent resistivity curves to show the effect of a variable surface layer. In all cases resistivity varies linearly with depth down to a depth of one unit. Material beneath this depth has constant resistivity. Electrode separation is plotted in depth units. Insets on each figure show the corresponding cross-sections, plotting true resistivity against depth.

To illustrate, consider curve *A* of Fig. 1. The true resistivity of material at the surface of the earth is taken as 0.4 units. Resistivity increases with depth, reaching a value of 1.0 at 0.5 depth units and 1.6 at 1.0 depth units. Below a depth of 1.0, all the material has very low resistivity (zero, for purposes of calculation). Curve *A* in the main part of Fig. 1 shows how apparent resistivity varies for this case as the electrode spacing is increased. To illustrate further, curve *E* of Fig. 4 corresponds to true resistivity of 1.2 units at the surface, 1.0 at a depth of 0.5 units, 0.8 at a depth of 1.0, and 1.5 for all depths greater than 1.0.

Apparent resistivity curves have been plotted logarithmically so that the shape of the curves becomes independent of the units, giving the curves wide validity. A certain drift-covered area, for example, shows a gradual decrease of resistivity from 230 ohm-meters at the surface to 150 ohm-meters at the bedrock surface, 275 ft down; bedrock resistivity is 800 ohm-meters. Curve *E* of Fig. 5 indicates that true resistivity decreases from 1.2 units at the surface to 0.8 at a depth of 1.0, then increases abruptly to a constant value of 4.0 units. Since resistivity and depth ratios are the same, this can be used to predict the field curve. For Fig. 5, multiply true resistivity and apparent resistivity by

H. M. MOONEY is Assistant Professor of Geophysics, University of Minnesota, Minneapolis, Minn.

Discussion on this paper, TP 3900L, may be sent (2 copies) to AIME before Jan. 31, 1954. Manuscript, March 29, 1954.

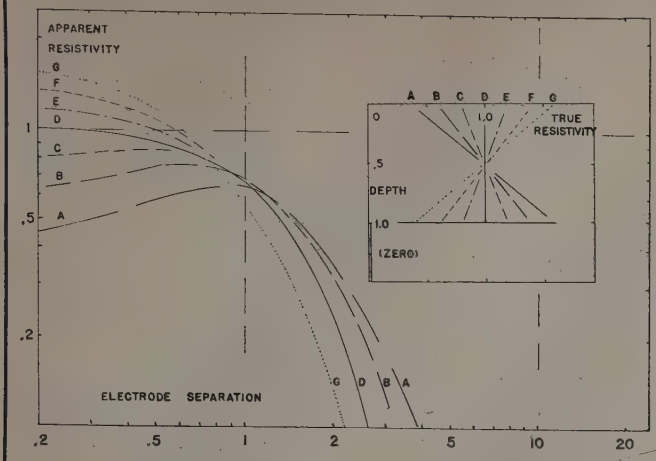


Fig. 1

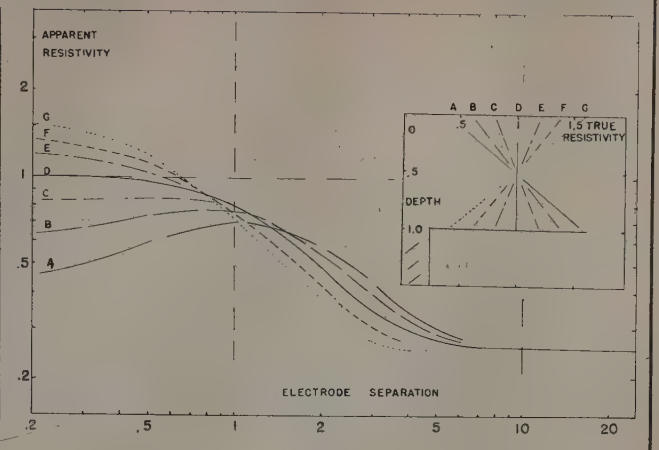


Fig. 2

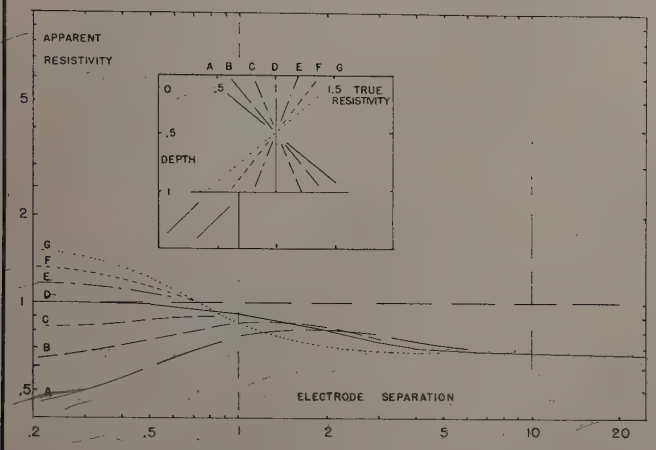


Fig. 3

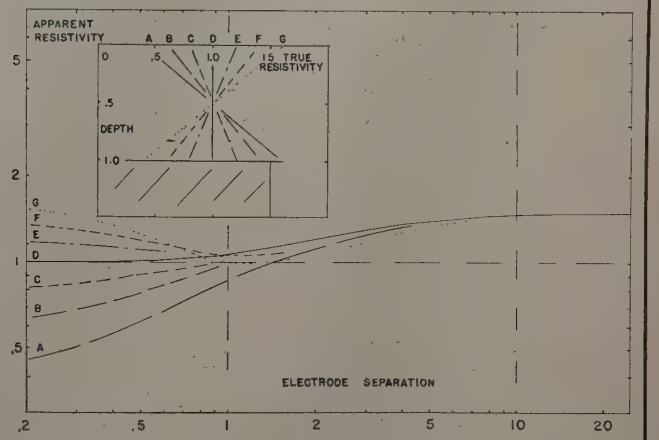


Fig. 4

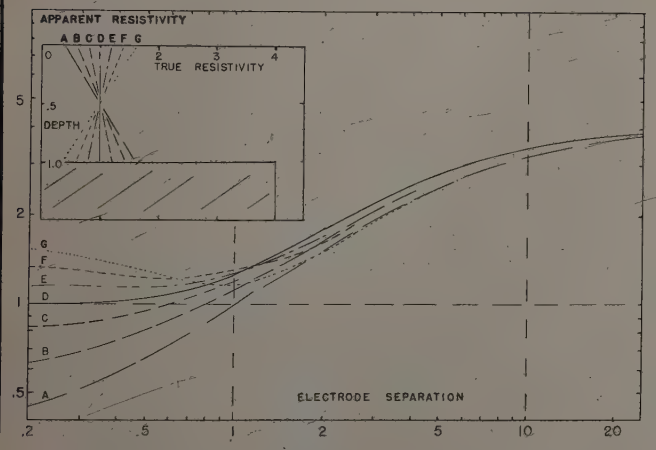


Fig. 5

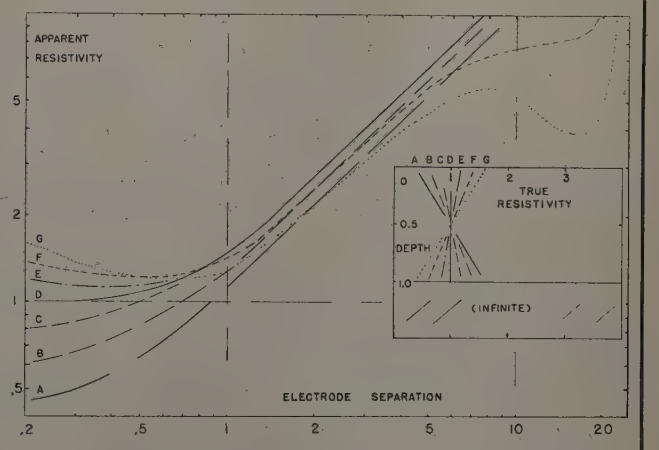


Fig. 6

Figs. 1-6 (reading across from upper left to lower right)—Apparent resistivity curves show the effect of a variable surface layer. In all cases resistivity varies linearly with depth. Material beneath this depth has constant resistivity. Electrode separation is plotted in depth units. Insets on each figure show the corresponding cross-sections, plotting true resistivity against depth.

230/1.2, labeling the new units ohm-meters; multiply depth and electrode separation by 275/1.0, labeling the new units feet. Curve *E* approximates the apparent resistivity data.

The preceding example used known geology to predict an apparent resistivity curve. The more usual situation, of course, is to infer geology from field data.

Curve *D* in all the figures represents the usual two-layer case of constant resistivity in the surface layer. This serves as a basis for comparison.

For those who wish a mathematical description of the curves, resistivity in the surface layer has been represented in the form $P_1 = K_1 + K_2Z$, where Z is depth and K's are constants. Curves *A*, *B*, *C*, etc. correspond to $(K_1, K_2) = (0.4, 1.2)$, $(0.6, 0.8)$, $(0.8, 0.4)$, $(1.0, 0.0)$, $(1.2, -0.4)$, $(1.4, -0.8)$, and $(1.6, -1.2)$. These particular combinations were chosen so that the average resistivity of the surface layer would be the same in all cases, namely 1.0. Figs. 1, 2, 3, etc. correspond to lower layer resistivities of 0, 1/4, 2/3, 3/2, 4, and infinity.

Calculations were carried out incidental to a larger project for computing four-layer curves; the mathematics will be described in the report of this project.[14] In summary, the surface layer was approximated by a series of five thin layers, each of constant resistivity. Surface potentials were evaluated with an electronic computer by numerical integration of an infinite integral. Although a general solution of this problem would be impossibly long, symmetry here permitted simplification.

When the curves are compared with the constant resistivity result in each figure (curve *D*), several conclusions may be drawn: 1—The major effect occurs for electrode separations less than the depth of the interface, i.e., separations less than 1.0. 2—For smaller separations, the curves asymptotically approach the true resistivity at the surface. 3—For larger separations, the deviation exceeds 10 pct only in severe cases. 4—For *high* resistivity in the lower layer, Figs. 4 to 6, *all* curves lie below curve *D* for larger electrode separations. 5—For *low* resistivity in the lower layer, Figs. 1 to 3, *all* curves cross curve *D*. 6—As would be expected, all curves are asymptotic to curve *D* for large separations, but this behavior is less apparent for high than for low resistivity in the lower layer.

Table I. Errors in Interpretation of Field Curves

Resistivity	P_1	Depth
Low resistivity in second layer, Figs. 1-3; increasing resistivity with depth, curves *A-C*	Too low	Too high
Low resistivity in second layer, Figs. 1-3; decreasing resistivity with depth, curves *E-G*	Too high	Too low
High resistivity in second layer, Figs. 4-6; increasing resistivity with depth, curves *A-C*	Too low	Too low
High resistivity in second layer, Figs. 4-6; decreasing resistivity with depth, curves *E-G*	Too high	Too high

For the larger electrode separations curves *F* and *G* of Fig. 6 show an effect of some mathematical interest. Inspection of the inset will show that for these cases resistivity drops to a very low value within the lower layer. In a slightly more extreme case, a thin perfect conductor can be imagined lying just above a thin perfect insulator. The presence of the insulator could then not be detected; the apparent resistivity curve would look something like *G* in Fig. 1. The beginnings of this tendency can be seen in curves *F* and *G* of Fig. 6. The phenomenon has no practical importance, since such extreme resistivity contrasts are most improbable geologically.

An interpreter faced with field curves of the sort shown in Figs. 1 to 6 might attempt to interpret them by matching to two-layer curves. His results would be in error as shown in Table I.

In all cases the matches are poor enough to arouse suspicion. A given field curve can be placed by inspection in one of the four categories given in Table I; directions of the suspected error are then known.

The curves of Figs. 1 to 6 could also be interpreted as three-layer problems; some provide a fairly good match. The trend here cannot be specified so well, for it will depend on the interpreter's judgment as to which match is most plausible. Experience shows that the depths are usually too low and that the interpreted surface resistivity approaches the true surface resistivity. This serious possibility for misinterpretation serves to emphasize a general rule for resistivity work: every area presents its own problems and requires careful preliminary work at control points before a field program can be attempted.

References

[1] H. M. Mooney: Depth Determinations by Electrical Resistivity. *Trans.* AIME (September 1954) **199**, p. 915.

[2] W. Stern: Resistivity Methods for Investigation of Tectonics and Underground Hydrology. *Beitr. Ang. Geoph.* (1933) **3**, p. 408. In German.

[3] R. F. Aldredge: The Effect of Dipping Strata on Earth Resistivity Determinations. *Colorado School of Mines Quarterly* (1937) **32**, p. 171.

[4] R. Trudu: Resistivity Curves for Two Layers with Plane Contact Slightly Inclined. *Riv. Geof. Appl.* (1952) **13**, p. 95. In Italian.

[5] T. Y. Berel'kovskiy and B. G. Zubanov: The Determination of an Inclined Contact by the Electrical Method. *Akad. Nauk. SSSR Izv. Ser. Geofiz.* (1951) **3**, p. 16. (Geophysical Abstracts 150, 13950). In Russian.

[6] M. Unz: Apparent Resistivity Curves for Dipping Beds. *Geophysics* (1953) **18**, p. 116.

[7] L. Cagniard: The Importance of the Phenomenon of Anisotropy in Electrical Sounding. *Inst. Phys. Globe Strasbourg Ann.* (1948) **4**, p. 3. In French.

[8] R. Maillet: Fundamental Equations of Electrical Prospecting. *Geophysics* (1947) **12**, p. 529.

[9] S. J. Pirson: Effect of Anisotropy on Apparent Resistivity Curves. *Bulletin of the American Association of Petroleum Geologists* (1935) **19**, p. 35.

[10] A. Belluigi: On the Effect of Anisotropy. *Beitr. Ang. Geoph.* (1934) **4**, p. 400. In German.

[11] R. Geneslay and F. Rouget: On Electrical Anisotropy and Pseudo-Anisotropy. *Proc.* 2me Cong. Mond. Petr., Paris (1937), p. 723. In French.

[12] W. G. Keck and W. F. Colby: The Depth Dependence of Earth Conductivity on Surface Potential Data. *Journal of Applied Physics* (1942) **13**, p. 17.

[13] A. F. Stevenson: On the Theoretical Determination of Earth Resistance from Surface Potential Measurements. *Physics* (1934) **5**, p. 114.

[14] H. M. Mooney and W. W. Wetzel: *Potentials about a Point Electrode and Apparent Resistivity Curves for a Four-Layered Earth.* Scheduled for 1955 publication, University of Minnesota Press.

ABSTRACT

Principles of fluid motion and turbulence which have been found to be of use in mixing and agitation problems are discussed, as well as suggested applications in extractive-metallurgy processes. Various types of impellers are described, together with other conditions that affect flow pattern and turbulence. The choice of equipment for particular requirements is considered, and equations for power input are given. Modern heavy-duty mixing and agitation equipment can take an increasingly important part in such extractive-metallurgy processes as solids handling, crystallizing and leaching, chemical operations, and flotation. Application of mixing and fluid-mechanics principles to extraction methods can lead to greater process rates and a resultant saving in time and money.

The article from which the above abstract is reprinted, *Fundamentals of Mixing and Agitation with Applications to Extractive Metallurgy*, by J. H. Rushton and L. H. Mahony, appears on pp. 1199-1206 of JOURNAL OF METALS, November 1954.

INDEX

Transactions, Mining Branch

January-December 1954, Vol. 199

A

Abrasion machine, evaluation of natural corundum ores ....Apr 402
Abrasives, natural ....Oct 1010
Activation Energies for the Decomposition of Limestone, Dolomitic Limestone, and Dolomite....July 730
Adsorption of a Mercaptan on Zinc Minerals ....Sep 925
Adsorption of Dodecylammonium Acetate on Hematite and Its Flotation Effect ....Dec 1196
Adsorption: Gibbs equation....May 519
oils by minerals....Jan 72
Young's equation ....May 519
Aeration, in flotation of pyrrhotite....Feb 215
Agglomeration, evaluation of sinter testing ....May 526
magnetite and hematite cubes fired in air ....Apr 414
solid state, in iron ore pellets....Apr 411
Alkali Reactivity of Natural Aggregates in Western United States. Discussion ....Nov 1114
Allen, T. M. and White, W. H.: *Copper Soil Anomalies in the Boundary District of British Columbia* Jan 49
Allentown Portland Cement Co., suspension preheating of dry pulverized materials ....Apr 407
Allis-Chalmers Manufacturing Co.: *Analysis of Variables in Rod Milling* Oct 1001
Latest Practice in Burning Cement and Lime in Europe....July 715
Aluminum, forms for concreting slusher drifts ....May 510
American Potash & Chemical Corp. Main Plant Cycle....Feb 203
American Smelting & Refining Co.: *Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes*....Oct 989
Structure and Mineralization at Silver Bell, Ariz. ....Nov 1095
Amine flotation of oxidized zinc ores Apr 417
Anaconda Copper Mining Co.: *Rock Bolting in Metal Mines of the Northwest* ....July 709
Sedimentary Rocks at Cananea, Sonora, Mexico, and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona ....June 628
Analysis of Mine Opening Failure by Means of Models. Discussion Nov 1102
Analysis of Variables in Rod Milling Oct 1001
Anderson, W. W., North, C. H., Lynd, L. E., and Sigurdson, H.: *Characteristics of Titaniferous Concentrates* ....Aug 817
Anomalies in copper soil, Boundary District, B. C....Jan 49
Appalachian Coals Inc.: *Trends in Coal Utilization and Their Effect on Coal Marketing* ....Mar 299
Application of Closed-Circuit TV to Conveyor and Mining Operations ....July 720
Arizona: geophysical discovery and development of the Pima mine Feb 197
sedimentary rocks, tentative correlation with at Cananea, Sonora, Mexico ....June 628
structure and mineralization at Silver Bell ....Nov 1095
uranium prospecting ....Feb 218
Asbestos, Jeffrey mine....May 510
Ash content in coal....May 506
Attwood Copper Mines Ltd.: copper exploration, British Columbia....Jan 49
Australia: uranium deposits....Sep 904

B

Ball mills, charging methods: Anaconda Copper Mining Co....June 625
Cananea Consolidated Copper Co. June 623
Chile Exploration Co....June 623
Climax Molybdenum Co....June 625
Idarado Mining Co....June 627
Phelps Dodge Corp....June 624, 626
Quemont Mining Corp....June 626
Ball wear, correlation between principal parameters affecting....June 642
Barite, Missouri, gravity surveys for residual deposits ....Jan 52
Battelle Memorial Institute: *Collection of Laboratory Dusts*....Aug 833
Properties of Nonmagnetic Taconites Affecting Concentration....June 635
Baum, John L.: *Magnetic Attraction of Stacked Drill Rods*....Dec 1203
Beach sands, characteristics of titaniferous concentrates from....Aug 817
Beneficiation: analysis of rod milling variables ....Oct 1001
ball wear, principal parameters affecting ....June 642
comminution ....Mar 313
fluorspar tailings, milling....May 542
manganese upgrading ....Mar 289
nonmagnetic taconites, properties affecting concentration ....June 635
pyrite-telluride flotation concentrates Dec 1181
tumbling mill capacity and power consumption as related to mill speed ....July 728
see also crushing, flotation, and concentration
Bennett, O. B.: *Diamond Drilling Problems at Rhokana*....Dec 1186
Bergstrom, B. H., Kirkland, T. G., Sollenberger, C. L., and Mitchell, Will, Jr.: *Analysis of Variables in Rod Milling*....Oct 1001
Bertholf, W. M. and Price, J. D.: *Improvements in Plant and Operations at Pueblo Coal Washery* Dec 1190
Bischoff, C. T.: *Testing for Copper and Zinc in Canadian Glacial Soils* Jan 57
Bituminous Coal Research Inc.: *Frontiers in Heat Extraction from the Combustion Gases of Coal* Mar 304
Blasting: Coplay Cement Mfg. Co....Jan 62
Jeffrey mine, Canadian Johns-Manville Co. Ltd....May 511
Michigan cut ....June 618
multi-burn cut ....June 617
Bond, Fred C.: *Discussion on a Physical Explanation of the Empirical Laws of Comminution*....Nov 1107
work index ....Oct 1004
Bonding: magnetite and hematite cubes fired in air....Apr 414
solid state, in iron ore pellets ....Apr 411
Boundary District, B. C.: copper soil anomalies ....Jan 49
Boyer, W. A.: *Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes*....Oct 989
Block Caving Practice at the Jeffrey Mine ....May 510
Borax and boric acid: production of, American Potash & Chemical Corp....Feb 203
Botanical Methods of Prospecting for Uranium ....Feb 217
Brandt, Robert E. and Cooke, Strathmore R. B.: *Solid State Bonding in Iron Ore Pellets*....Apr 411
Brazil: lithium production....Sep 897
Brazing: tungsten carbide tipped drill steel ....Mar 294
Breckenridge, Robert N. and Pollish, Lloyd: *Rock Bolting in Metal Mines of the Northwest*....July 709
British Columbia: copper soil anomalies in Boundary District ....Jan 49
Brown Iron Ore Resources of Missouri Jan 63
Burwell, E. B., Jr., and Nesbitt, R. H: *NX Borehole Camera*....Aug 805

C

California: potash, Searles Lake....Feb 203
Canada: abrasives, natural....Oct 1010
asbestos mining ....May 510
Canadian Dept. of Mines and Technical Surveys, Ottawa: *Evaluating the Performance of a Cleaning Unit* Oct 1015
Canadian glacial soils, testing for copper and zinc....Jan 57
Canadian Johns-Manville Co. Ltd.: *Block Caving Practice at the Jeffrey Mine* ....May 510
Canadian Mines Branch, Ottawa: *Natural Abrasives in Canada*....Oct 1010
Cananea Consolidated Copper Co.: *Sedimentary Rocks and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona* ....June 628
Cannon, Helen L.: *Botanical Methods of Prospecting for Uranium* ....Feb 217
Carbon-bearing deposits of the Western Hemisphere ....Sep 908
Carl, Howard, Jaffe, Howard W., and Hockman, Arthur: *New Techniques for Evaluating Natural Corundum Ores* ....Apr 402
Carlton mill, Golden Cycle Corp....Dec 1181
Cement: decomposition of limestone July 730
modern shaft kilns....July 715
suspension preheating of....Apr 407
Centrifuges, types and applications....May 534
Chain Belt Co. of Milwaukee: pump-crete machine ....Apr 393
Chalcopyrite, British Columbia....Jan 49
Chang, C. S.: *Substituted Starches in Amine Flotation of Iron Ore* Sep 922
Chang, C. S., Cooke, Strathmore R. B., and Iwasaki, Iwao: *Flotation Characteristics of Pyrrhotite with Xanthates* ....Feb 209
Change to Rotary Blasthole Drilling in Limestone Increases Footage, Cuts Time, Saves Manpower Aug 809
Characteristics of Titaniferous Concentrates ....Aug 817
discussion ....Nov 1116
Cheasley, T. C., Forrester, J. D., and Sarapuu, Erich: *Underground Electrocarbonization of Coal and Related Hydrocarbons* ....Sep 908
Clark, Edward L. and Muilenburg, Garrett A.: *Brown Iron Ore Resources of Missouri*....Jan 63
Clark, George B.: *Discussion on A Physical Explanation of the Empirical Laws of Comminution*....Nov 1106
Relationship of Geology to Underground Mining Methods....Aug 812
Classification: coal ....Dec 1190
fluorspar tailings ....May 542
liquid-solid cyclone ....June 620
manganese ore ....Mar 291
Classifiers, types and applications....May 534
Cleaning Various Coals in a Drum-Type Dense-Medium Pilot Plant. Discussion ....Nov 1110
Clemmer, J. Bruce, Rosenbaum, J. B., and Schack, C. H.: *Discussion on Manganese Upgrading at Three Kids Mine, Nevada*....Nov 1105
Cleveland-Cliffs Iron Co.: *Mather Mine Uses Pipeline Concrete in Underground Operations* ....Apr 393
Coal: ash content ....May 506
bituminous, flotation of....Jan 73, 74
classification ....Dec 1190
cleaning units, evaluation of....Oct 1015
crushing ....Dec 1193
dewatering ....Dec 1190
heat extraction from combustion gases Mar 304
longwall mining....July 701, Aug 828
marketing ....Mar 299
Oaxaca fields, Mexico, exploration of May 505
oil spraying ....July 724
oxidation of on flotation properties Apr 396
pitching seams, mining of....Aug 825
underground electrocarbonization Sep 909
utilization ....Mar 299
Coleman Collieries Ltd.: *Productivity in Mining Pitching Seams of the Canadian Rockies* ....Aug 825
Collection of Laboratory Dusts....Aug 833
Colorada copper orebody, Cananea, Mexico ....May 500
Colorado Fuel & Iron Corp.: *Improvements in Plant and Operations at Pueblo Washery*....Dec 1189
Colorado Plateau, uranium: origin of deposits ....Sep 907
prospecting ....Feb 218
Colorimetric testing: copper and zinc Jan 49, 57
mill product purity of transparent minerals ....Sep 919
Commercial Synthesis of Star Sapphires and Star Rubies....Jan 78
Comminution, physical explanation of empirical laws ....Mar 313
Concentration: affected by properties of nonmagnetic taconites ....June 635
fluorspar ....May 544
Concrete: aluminum forms for in underground mining ....May 510
pipelines in underground operations Apr 393
wetting agents in....Apr 396
Continental Sales & Equipment Co.: *Properties of Nonmagnetic Taconites Affecting Concentration* June 635
Cooke, Strathmore R. B. and Brandt, Robert E.: *Solid State Bonding in Iron Ore Pellets* ....Apr 411
Cooke, Strathmore R. B., Chang, C. S., and Iwasaki, Iwao: *Flotation Characteristics of Pyrrhotite with Xanthates* ....Feb 209

Coplay Cement Manufacturing Co.: *Trackless Development of an Inclined Limestone Deposit*....Jan 61
Copper: association of deposits with granites and porphyries.....May 499
closed-circuit grinding of.....June 620
Mineral Hill, Arizona .....Feb 198
testing for in Canadian glacial soils Jan 57
Copper Soil Anomalies in the Boundary District of British Columbia Jan 49
Copper-activated pyrrhotite .....Feb 209
Correlation Between Principal Parameters Affecting Mechanical Ball Wear .....June 642
Cortes-Obregon, Salvador and Toron, Luis: *Exploration of the Oaxaca Coal Fields in Southern Mexico* .....May 505
Corundum: deposits in Canada.....Oct 1010
National Stockpile Materials Specifications .....Apr 406
new techniques for evaluating.....Apr 402
Courtright, James H. and Richard, Kenyon, E.: *Structure and Mineralization at Silver Bell, Ariz.* Nov 1095
Crushing: coal .....Dec 1193
Cyclones: classifiers for manganese ore Mar 291
liquid-solid, in closed-circuit grinding of concentrates .....June 620

D

Daniel C. Jackling Award: Presentation May 497
de Bruyn, P. L., Overbeek, J. Th., and Schuhmann, R., Jr.: *Flotation and the Gibbs Adsorption Equation* .....May 519
Department of the Army, Office of the Chief of Engineers: *NX Borehole Camera* .....Aug 805
Depth Determinations by Electrical Resistivity .....Sep 915
Development and Construction of a Ground-Water Supply .....Nov 1093
Diamonds, industrial .....Oct 1010
Diamond Drilling Problems at Rhokana Dec 1186
Diamond Power Specialty Corp.: *Application of Closed-Circuit TV to Conveyor and Mining Operations* July 720
Discussion: Mining .....Nov 1101
Minerals Beneficiation .....Nov 1103
Coal .....Nov 1108
Industrial Minerals .....Nov 1114
Dithizone, used in testing for copper and zinc .....Jan 49, 57
Dolomite and dolomitic limestone, activation energies for the decomposition of .....July 730
Dominion Steel and Coal Corp. Ltd.: *Longwall Mining and Mechanization, with Special Reference to Nova Scotia*.....July 701
Dorr Co.: *Guide to the Proper Application of Classifiers*.....May 534
Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates .....June 620
tests with FluoSolids reactor at Golden Cycle Corp. .....Dec 1182
Dosco miner .....July 706
Doxey, Frank: *Longwall Mining and Mechanization, with Special Reference to Nova Scotia*...July 701
Drill rods, magnetic attraction when stacked .....Dec 1202
Drills, core .....Dec 1187, 1207
Drills and drilling: equipment.....May 507
fanholes in top slicing at Kiruna mine, Sweden .....June 613
ground-water control, equipment June 629
Jeffrey mine .....May 514
pneumatic hammer, used in Sweden June 616
rotary blasthole.....May 510, Aug 809
sectional steel at Jeffrey mine.... June 615
Sweden, use of flexible steel.....June 617
wagon, at Boliden mine, Sweden...June 617
Drilling Practice in Swedish Mining June 613
Dust collection: laboratories.....Aug 833
Three Kids mine, Nevada.....Mar 293

E

Effect of a Variable Surface Layer on Apparent Resistivity Data...Dec 1210
Effects of Oxidation of Coals on Their Flotation Properties .....Apr 396
Elder, James L.: *Discussion on Underground Electrocarbonization of Coal and Related Hydrocarbons* Nov 1108
Electrical resistivity: depth determinations by .....Sep 915
effects of a variable surface layer on data .....Dec 1210
Electrotherm Research Corp.: *Underground Electrocarbonization of Coals and Related Hydrocarbons* Sep 908
Engelhart, G. K.: *Suspension Preheating of Dry Pulverized Materials* Apr 407
Equipment: classifying, manganese ore Mar 291
diamond drilling .....Dec 1186
diesel locomotive .....July 705
drill exploration of Pima mine area Feb 201
drilling .....May 507, 510
hydrogen reducibility of sinters..May 528
pneumatic placers for underground concrete .....Apr 393
scrapers .....June 618
soil testing .....Jan 49
trackless, in development of inclined limestone deposit .....Jan 61
Estudios Geologic-Mineros, Banco de Mexico, S. A.: *Exploration of the Oaxaca Coal Fields in Southern Mexico* .....May 505
Evaluating the Performance of a Cleaning Unit .....Oct 1015
Evaluation of Sinter Testing.....May 527
Everhart, D. L.: *Origin of Uranium Deposits. A Progress Report*....Sep 904
Ewers, W. E.: *Discussion on Flotation and the Gibbs Adsorption Equation* .....Nov 1103
Exploration of the Oaxaca Coal Fields in Southern Mexico .....May 505
discussion .....Nov 1113
Extractive metallurgy, applications of mixing and agitation to. Abstract .....Dec 1212

F

Fatty acids, in flotation of zinc ores Apr 416
Federal Coal Mine Safety Act.....Jan 76
Finland Institute of Technology: *Correlation Between Principal Parameters Affecting Mechanical Ball Wear* .....June 642
Tumbling Hill Capacity and Power Consumption as Related to Mill Speed .....July 728
Firing of magnetite pellets.....Apr 411
Fisher, H. M., Sun, S. C., and Snow, R. E.: *Rapid Estimation of Mill Product Purity by Transparency Measurement* .....Sep 919
Florida: phosphate reserves, source of fluorine .....Oct 995
Flotation: coal, bituminous .....Apr 396
fluorspar .....May 542
hematite, adsorption of dodecylammonium acetate on .....Dec 1196
hypothesis for coals, carbons, and hydrocarbon minerals .....Jan 67
iron ore .....Sep 922, Dec 1181
manganese ore .....Mar 291
theory of molecular interaction.....Feb 221
zinc, minerals, adsorption of a mercaptan on .....Sep 925
ores, oxidized .....Apr 416
Flotation and the Gibbs Adsorption Equation .....May 519
correction .....Aug 808
discussion .....Nov 1103
Flotation Characteristics of Pyrrhotite with Xanthates .....Feb 209
Flotation of Oxidized Zinc Ores.....Apr 416
Flotation Theory: Molecular Interaction Between Frothers and Collectors at Solid-Liquid-Air Interfaces .....Feb 221
Fluorine, from phosphate rock.....Oct 994
Fluorspar, flotation of .....May 542
FluoSolids, reactor, Golden Cycle Corp. Dec 1811
Forbes, J. J.: *Federal Coal Mine Safety Act* .....Jan 76
Formanek, V., Raffinot, P., Rey, M., and Sitia, G.: *Flotation of Oxidized Zinc Ores* .....Apr 416
Forrester, J. D., Cheasley, T. C., and Sarapuu, Erich: *Underground Electrocarbonization of Coal and Related Hydrocarbons* .....Sep 908
France: milling plants, oxidized zinc ores .....Apr 418
French Morocco: milling plants, oxidized zinc ores .....Apr 418
Frondel, Clifford: *Commercial Synthesis of Star Sapphires and Star Rubies* .....Jan 78
Frontiers in Heat Extraction from the Combustion Gases of Coal....Mar 304
discussion .....Nov 1112
Frothing agents, in flotation of zinc ores Apr 418
Fuller Co.: *Suspension Preheating of Dry Pulverized Materials* ....Apr 407
Furnaces: electric tube .....Apr 412
firing specimens of magnetite pellets Apr 411

G

Garnet, world market.....Oct 1012
Garrow rod-charging machine.....June 622
Gaudin, A. M. and Harris, D. L.: *Adsorption of a Mercaptan on Zinc Minerals* .....Sep 925
Gaudin, A. M. and Morrow, J. G.: *Adsorption of Dodecylammonium Acetate on Hematite and Its Flotation Effect* .....Dec 1196
Genetic Relations Between Granites, Porphyries, and Associated Copper Deposits .....May 499
Geological Survey, creation of in 1879 Apr 421
Geology and geophysics: Cananea, sedimentary rocks, tentative correlation with sections at Bisbee and Swisshelm Mountains, Arizona .....June 628
copper, Colorada orebody, Cananea, Mexico .....May 500
deposits near Chuquicamata, Chile May 500
soil anomalies, British Columbia, testing for .....Jan 49, 57
discovery and development of the Pima mine, Arizona.....Feb 197
electrical resistivity, depth determinations by .....Sep 915
effect of a variable surface layer on data .....Dec 1210
factors affecting mining operations Aug 814
genetic relations between granites, porphyries, and associated copper deposits .....May 499
gravity surveys for residual barite deposits in Missouri.....Jan 52
hydrocarbons, near-surface, and petroleum accumulations at depth Dec 1205
magnetic attraction of stacked drill rods .....Dec 1203
Oaxaca coal fields, Mexico, exploration of .....May 505
scintillation counter in search for oil July 727
Silver Bell, Ariz., structure and mineralization at .....Nov 1095
theories of ore deposition.....May 499
titaniferous concentrates, from beach sands, characteristics of .....Aug 817
uranium, botanical methods of prospecting for .....Feb 217
origin of deposits .....Sep 904
zinc, soil anomalies, British Columbia, testing for .....Jan 57
Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project .....Feb 197
Germany: suspension preheating of dry pulverized materials .....Apr 407
Gold: flotation of concentrates.....Dec 1181
Mineral Hill, Ariz.....Feb 198
Golden Cycle Corp., Carlton Mill: *Operation of FluoSolids Roaster at Golden Cycle* .....Dec 1181
Granites, porphyries, and associated copper deposits, genetic relations May 499
Grant, B. E.: *Discussion on Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes* .....Nov 1101
Grantham, D. R.: *Discussion on Characteristics of Titaniferous Concentrates* .....Nov 1116
Graton-Vanderwilt polishing machine Apr 413
Gravity Surveys for Residual Barite Deposits in Missouri.....Jan 52
Griffen, John: *Discussion on Cleaning Coals in a Drum-Type Dense-Medium Pilot Plant*.....Nov 1110
Ground Water Control in Underground Mining .....June 632
Groundwater supply, construction of Nov 1093
Guide to the Proper Application of Classifiers .....May 534

H

Hanna Coal Co.: *Oil Spraying at the Georgetown Preparation Plant* July 724
Safety in the Mechanical Mining of Coal .....May 524
Hardy, Carroll F.: *Trends in Coal Utilization and Their Effect on Coal Marketing* .....Mar 299
Harris, D. L. and Gaudin, A. M.: *Adsorption of a Mercaptan on Zinc Minerals* .....Sep 925
Harvard University: *Commercial Synthesis of Star Sapphires and Star Rubies* .....Jan 78
Haulage: Dominion Steel & Coal Corp. Ltd., Cape Breton Coal Operations, Sydney, Nova Scotia July 706
Hanna Coal Co.....May 524
Heat extraction from combustion gases of coal .....Mar 304
Heinrichs, Walter E., Jr., Thurmond, Robert E., and Spaulding, E. D.: *Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project*....Feb 197
Hematite: Adsorption of dodyclammonium acetate on.....Dec 1196
in plant sinters .....May 530
Hill, W. L. and Jacob, K. D.: *Phosphate Rock as an Economic Source of Fluorine* .....Oct 994
Hitzrot, H. W.: *Guide to the Proper Application of Classifiers*.....May 534
Hockman, Arthur, Jaffe, Howard W., and Carl, Howard: *New Techniques for Evaluating Natural Corundum Ores* .....Apr 402
Hoisting ropes, safety factor.....Oct 989
Horvitz, Leo: *Near-Surface Hydrocarbons and Petroleum Accumulations at Depth* .....Dec 1205
Hudson Bay Mining & Smelting Co. Ltd.: *Manufacture of Tungsten Carbide Tipped Drill Steel*...Mar 294
Hukki, R. T.: *Correlation Between Principal Parameters Affecting Mechanical Ball Wear*.....June 642
Tumbling Mill Capacity and Power Consumption as Related to Mill Speed .....July 728
Humboldt suspension preheater.....Apr 407
Hydrocarbon surveys .....Dec 1205
Hydrology .....June 632, Nov 1093
Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals .....Jan 67

I

Idaho: phosphate reserves, source of fluorine .....Oct 995
Improvements in Plant and Operations at Pueblo Coal Washery.....Dec 1190

Iron ore: flotation of....Sep 922, Dec 1196
brown ore resources, Missouri....Jan 63
nonmagnetic taconites, properties affecting concentration....June 635
pellets, solid state bonding in....Apr 411
sinter testing....May 527
Iron oxide materials, analysis of....Apr 411
Italy: flotation of oxidized zinc ores Apr 416
Iwasaki, Iwao, Cooke, Strathmore R. B., and Chang, C. S.: *Flotation Characteristics of Pyrrhotite with Xanthates*....Feb 209

J

Jacob, K. D. and Hill, W. L.: *Phosphate Rock as an Economic Source of Fluorine*....Oct 994
Jaffe, Howard W., Carl, Howard, and Hockman, Arthur: *New Techniques for Evaluating Natural Corundum Ores*....Apr 402
Janelid, Ingvar: *Drilling Practice in Swedish Mining*....June 613
Janes, T. H.: *Natural Abrasives in Canada*....Oct 1010
Jeffrey mine, block caving practice May 510
Jenkins, R. W.: *Trackless Development of an Inclined Limestone Deposit*....Jan 61
Jensen, Owen F.: *Development and Construction of a Ground-Water Supply*....Nov 1093
Johnson, E. C. and Lewis, F. M.: *Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates*....June 620
Johnson, Oscar: *Methods of Charging Rods and Balls into Grinding Mills*....June 622

K

Kaiser, Elmer R.: *Frontiers in Heat Extraction from the Combustion Gases of Coal*....Mar 304
Kececioglu, Dimitri: *Discussion on A Physical Explanation of the Empirical Laws of Comminution* Nov 1106
Keil, Howard R.: *Operation of FluoSolids Roaster at Golden Cycle* Dec 1181
Kilns: European practice....July 715
manganese nodulizing....Mar 292
suspension preheating....Apr 407
Kinelski, E. H., Morrissey, H. A., and Powers, R. E.: *Evaluation of Sinter Testing*....May 527
Kirkland, T. G., Bergstrom, B. H., Mitchell, Will, Jr., and Sollenberger, C. L.: *Analysis of Variables in Rod Milling*....Oct 1001

L

Langston, Benny and Stephens, Frank M., Jr: *Collection of Laboratory Dusts*....Aug 833
Latest Practice in Burning Cement and Lime in Europe....July 715
Lead: Mineral Hill, Arizona....Feb 198
Leasing of Government Potash Lands Apr 421
Leja, J. and Schulman, J. H.: *Flotation Theory: Molecular Interactions Between Frothers and Collectors at Solid-Liquid-Air Interfaces*....Feb 221
Lellep, O. G., inventor of Lepol kiln Apr 407
Latest Practice in Burning Cement and Lime in Europe....July 715
Leonardi, M. L.: *American Potash & Chemical Corp. Main Plant Cycle*....Feb 203
Lewis, F. M. and Johnson, E. C.: *Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates*....June 620
Limestone: activation energies for decomposition of....July 730
rotary blasthole drilling in....Aug 809
Limonite, Missouri....Jan 63
Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates....June 620
Lithium concentrates and lithium carbonates, production of, American Potash & Chemical Corp. Feb 203
Lithium—Northeast Brazil is Potential Source....Sep 897
Locomotives, diesel, in mine haulage July 705
Longwall Mining and Mechanization, with Special Reference to Nova Scotia....July 701
discussion....Nov 1109
Lynd, L. E., Sigurdson, H., North, C. H., and Anderson, W. W.: *Characteristics of Titaniferous Concentrates*....Aug 817

M

M. A. Hanna Co.: *Ground Water Control in Underground Mining* June 632
Magnetic Attraction of Stacked Drill Rods....Dec 1203
Magnetite: in plant sinters....May 530
strength of fired pellets....Apr 411
taconite concentration....June 635
Mahon, R. C.: *Ground Water Control in Underground Mining*....June 632
Mahony, L. H. and Rushton, J. H.: *Fundamentals of Mixing and Agitation with Applications to Extractive Metallurgy*. Abstract Dec 1212
Manganese Inc.: *Manganese Upgrading at Three Kids Mine, Nevada* Mar 289
Manganese Upgrading at Three Kids Mine, Nevada....Mar 289
discussion....Nov 1105
Manufacture of Tungsten Carbide Tipped Steel....Mar 294
Manual on Rock Blasting, reference to June 618, 619
Market situations: abrasives, natural Oct 1012
coal....Mar 299
fluorine....Oct 996, 999
star sapphires and star rubies....Jan 78
Massachusetts Institute of Technology: *Adsorption of a Mercaptan on Zinc Minerals*....Sep 925
Adsorption of Dodecylammonium Acetate on Hematite and Its Flotation Effect....Dec 1196
Physical Explanation of the Empirical Laws of Comminution....Mar 313
Mather Mine Uses Pipeline Concrete in Underground Operations....Apr 393
Mather, W. B.: *Lithium—Northeast Brazil Is Potential Source*....Sep 897
McCarroll, S. J.: *Manganese Upgrading at Three Kids Mine, Nevada* Mar 289
Mechanical ball wear, correlation between principal parameters affecting....June 642
Meger, A. F.: *Oil Spraying at the Georgetown Preparation Plant*....July 724
Mellon Institute: *Evaluation of Sinter Testing*....May 527
Merritt, W. L.: *Discussion on Alkali Reactivity in Western United States*....Nov 1115
Mesabi taconites, properties of....June 636
Methods of Charging Rods and Balls into Grinding Mills....June 622
Mexico: copper deposits at Cananea May 500
Oaxaca coal fields, exploration of May 505
Michigan: ground water control at Iron River....June 632
Milling: classifiers, application....May 534
closed-circuit grinding of concentrates June 620
fluorspar tailings....May 542
methods of charging rods and balls June 622
(see also Beneficiation)
Milling Kentucky Fluorspar Tailings May 542
Mine & Smelter Supply Co.: *Methods of Charging Rods and Balls into Grinding Mills*....June 622
Mining methods: aluminum forms for concreting slusher drifts....May 510
blasting, Coplay Cement Manufacturing Co....Jan 62
Michigan cut....June 618
multiburn cut....June 617
block caving....May 510
concreting, at Jeffrey mine, Canadian Johns-Manville Co. Ltd....May 512
at Mather mine, Cleveland-Cliffs Iron Co....Apr 393
crosscuts....May 510
cut-and-fill....June 617, Aug 813
drawpoints....May 510
fanholes in top slicing....June 617
flexible drill steel used in Sweden June 614
groundwater control....June 628
haulage system, Hanna Coal Co. May 524
hoisting ropes, safety factor....Oct 989
longwall mining and mechanization July 701
pilot drifts....May 510
pipeline concrete....Apr 393
pneumatic stowing....July 705
relationship of geology to....Aug 749
rock bolting....July 704, 709
scrapers for small tunnels....June 618
slusher drift development....May 510
steel ladders used for driving raises, Sweden....June 618
stoping....Aug 813, Dec 1189
undercutting technique at Jeffrey mine May 513
Missouri: brown iron ore resources Jan 63
gravity surveys for residual barite deposits....Jan 52
Missouri Geological Survey: *Brown Iron Ore Resources of Missouri*..Jan 63
Mitchell, Will, Jr., Sollenberger, C. L., Kirkland, T. G., and Bergstrom, B. H.: *Analysis of Variables in Rod Milling*....Oct 1001
Molybdenum, Mineral Hill, Ariz....Feb 198
Monroe, D. L., Waller, H. H., and Smyth, D. P. R.: *Block Caving at Jeffrey Mine*....May 510
Montana: mineralization periods at Butte....May 503
phosphate reserves, source of fluorine Oct 995
Mooney, Harold M.: *Depth Determinations by Electrical Resistivity* Sep 915
Effect of a Variable Surface Layer on Apparent Resistivity Data....Dec 1210
Morrissey, H. A., Kinelski, E. H., and Powers, R. E.: *Evaluation of Sinter Testing*....May 527
Morrow, J. G. and Gaudin, A. M.: *Adsorption of Dodecylammonium Acetate on Hematite and Its Flotation Effect*....Dec 1196
Muilenburg, Garrett A. and Clark, Edward L.: *Brown Iron Ore Resources of Missouri*....Jan 63
Mulchay, Roland B. and Velasco, J. Ruben: *Sedimentary Rocks at Cananea, Sonora, Mexico, and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona* June 628

N

National Bureau of Standards: laboratory abrasion machine....Apr 402
New Techniques for Evaluating Natural Corundum Ores....Apr 402
National Lead Co.: *Characteristics of Titaniferous Concentrates*....Aug 817
Gravity Surveys for Residual Barite Deposits in Missouri....Jan 52
Natural Abrasives in Canada....Oct 1010
Near-Surface Hydrocarbons and Petroleum Accumulations at Depth Dec 1205
Nesbitt, R. H. and Burwell, E. B., Jr.: *NX Borehole Camera*....Aug 805
New Jersey Zinc Co.: *Magnetic Attraction of Stacked Drill Rods*....Dec 1202
New Techniques for Evaluating Natural Corundum Ores....Apr 402
New York State: garnet production Oct 1012
North, C. H., Lynd, L. E., Sigurdson, H., and Anderson, W. W.: *Characteristics of Titaniferous Concentrates*....Aug 817
Nova Scotia, longwall mining and mechanization....July 701
NX Borehole Camera....Aug 805

O

O'Hara, T. A.: *Manufacture of Tungsten Carbide Drill Steel*....Mar 294
Oil Spraying at the Georgetown Preparation....July 724
Ontario: uranium deposits....Sep 904
Operation of FluoSolids Roaster at Golden Cycle....Dec 1181
Origin of Uranium Deposits. A Progress Report....Sep 904
Overbeek, J. Th., de Bruyn, P. L., and Schuhmann, R., Jr.: *Flotation and the Gibbs Adsorption Equation*....May 519
Oxidation of coals, effects on flotation properties....Apr 396
Oxide zinc minerals, recovery from sulphide ores....Apr 416

P

Pennsylvania State University: *Activation Energies for the Decomposition of Limestone, Dolomitic Limestone, and Dolomite*....July 730
Effects of Oxidation of Coals on Their Flotation Properties....Apr 396
Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals....Jan 67
Rapid Estimation of Mill Product Purity by Transparency Measurement....Sep 919
Phosphate Rock as an Economic Source of Fluorine....Oct 994
Physical Explanation of the Empirical Laws of Comminution....Mar 313
discussion....Nov 1106
Pima Mining Co.: *Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project* Feb 198
Pipeline concrete in underground operations....Apr 393
Plant analysis in prospecting for uranium....Feb 217
Pneumatic placers for underground concrete....Apr 393
Pollish, Lloyd and Breckenridge, Robert N.: *Rock Bolting in Metal Mines of the Northwest*....July 709
Porphyries, granites, and associated copper deposits, genetic relations of May 499
Potash: leasing of government lands Apr 421
production of, American Potash & Chemical Corp....Feb 203
Powers, R. E., Morrissey, H. A., and Kinelski, E. H.: *Evaluation of Sinter Testing*....May 527
Precipitator, Cottrell, at Golden Cycle Corp....Dec 1183, 1184
Price, John D.: *Discussion on Exploration of the Oaxaca Coal Fields in Southern Mexico*....Nov 1113
Price, J. D. and Berthoff, W. M.: *Improvements in Plant and Operations at Pueblo Coal Washery* Dec 1190
Productivity in Mining Pitching Seams of the Canadian Rockies....Aug 825
Properties of Nonmagnetic Taconites Affecting Concentration....June 635
Pumicite, sources....Oct 1014
Pumps and pumping: centrifugal, in ground water control....June 634
Moyno pump, reactor fed by....Dec 1181
pumpcrete machine, at Mather mine Apr 393
Pyrrhotite: flotation characteristics with xanthates....Feb 209

R

Raffinot, P., Rey, M., Sitia, G., and Formanek, V.: *Flotation of Oxidized Zinc Ores*....Apr 416

Rapid Estimation of Mill Product Purity by Transparency Measurement Sep 919
Reactor, FluoSolids, illustrated Dec 1183
Relationship of Geology to Underground Mining Methods Aug 812
Rey, M., Sitia, G., Raffinot, P., and Formanek, V.: *Flotation of Oxidized Zinc Ores* Apr 416
Reynolds, Dexter H.: *Discussion on Alkali Reactivity in Western United States* Nov 1114
Rhokana Corp. Ltd.: *Diamond Drilling Problems at Rhokana* Dec 1186
Richard, Kenyon E. and Courtright, James H.: *Structure and Mineralization at Silver Bell, Ariz.* Nov 1095
Rock Bolting in Metal Mines of the Northwest July 709
Rod milling, analysis of variables Oct 1001
Rod mills, charging methods: Anaconda Copper Mining Co. June 625
Chile Exploration Co. June 623
International Nickel Co. June 622
Oliver Iron Mining Co. June 622
Quemont Mining Corp. June 625
St. Joseph Lead Co. June 622
Union Minière du Haut Katanga June 622
Waite Amulet Mines Ltd. June 627
Rosenbaum, J. B., Clemmer, J. Bruce, and Schack, C. H.: *Discussion on Manganese Upgrading at Three Kids Mine, Nevada* Nov 1105
Rotary kiln waste gases, effective use of Apr 407
Royal Institute of Technology, Stockholm, Sweden: *Drilling Practice in Swedish Mining* June 613
Rubies: natural corundum Apr 402
star, commercial synthesis of Jan 78
Rushton, J. H. and Mahony, L. H.: *Fundamentals of Mixing and Agitation with Applications to Extractive Metallurgy* Dec 1212

S

Safety Factor Characteristic Curves. Their Application to Mine Hoisting Ropes Oct 989
discussion Nov 1101
Safety in the Mechanical Mining of Coal May 524
Sales, Reno H.: *Genetic Relations Between Granites, Porphyries, and Associated Copper Deposits.* The Daniel C. Jackling Lecture, February 1954 May 499
Sapphires: blue and yellow, natural corundum Apr 402
star, commercial synthesis of Jan 78
Sarapuu, Erich, Cheasley, T. C., and Forrester, J. D.: *Underground Electrocarbonization of Coal and Related Hydrocarbons* Sep 908
Saskatchewan: uranium deposits Sep 904
Schack, C. H., Rosenbaum, J. B., and Clemmer, J. Bruce: *Discussion on Manganese Upgrading at Three Kids Mine, Nevada* Nov 1105
Scharon, LeRoy and Uhley, Robert P.: *Gravity Surveys for Residual Barite Deposits in Missouri* Jan 52
Schuhmann, R., Jr., Overbeek, J. Th., and de Bruyn, P. L.: *Flotation and the Gibbs Adsorption Equation* May 519
Schulman, J. H. and Leja, J.: Flotation Theory: *Molecular Interactions Between Frothers and Collectors at Solid-Liquid-Air Interfaces* Feb 221
Schuster, W. J.: *Safety in the Mechanical Mining of Coal* May 524
Scintillation Counter in the Search for Oil Dec 1205
Scott, Donald W. and Wesner, Adam: *Properties of Nonmagnetic Taconites Affecting Concentration* June 635
Scrapers, for small tunnels June 618
Searls, Fred, Jr.: *Presentation*, Daniel C. Jackling Award May 497
Sedimentary Rocks at Cananea, Sonora, Mexico, and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona June 628
Shaft sinking: Jeffrey mine, Canadian Johns-Manville Co. Ltd. May 518
Mather mine, Cleveland-Cliffs Iron Co. Apr 393
Shaw, M. C. and Walker, D. R.: *A Physical Explanation of the Empirical Laws of Comminution* Mar 313
Sierrita Mountains, Pima mine district, Arizona Feb 198
Sigurdson, H., Lynd, L. E., North, C. H., and Anderson, W. W.: *Characteristics of Titaniferous Concentrates* Aug 817
Silenium-indicator plants in uranium districts Feb 219
Silver, Mineral Hill, Ariz. Feb 198
Sinclair Coal Co.: *Underground Electrocarbonization of Coal and Related Hydrocarbons* Sep 908
Sitia, G., Rey, M., Raffinot, P., and Formanek, V.: *Flotation of Oxidized Zinc Ores* Apr 416
Slusher drift development May 510
Smith, H. I.: *Leasing of Government Potash Lands* Apr 421
Smithsonite, flotation of Apr 416
Smyth, D. P. R., Waller, H. H., and Monroe, D. L.: *Block Caving at the Jeffrey Mine* May 510
Snow, R. E., Fisher, H. M., and Sun, S. C.: *Rapid Estimation of Mill Product Purity by Transparency Measurement* Sep 919
Società Mineraria è Metallurgica di Pertusola Genoa: *Flotation of Oxidized Zinc Ores* Apr 416
Société Minerais et Metaux: *Flotation of Oxidized Zinc Ores* Apr 416
Société Minière et Metallurgique de Penarroya Paris: *Flotation of Oxidized Zinc Ores* Apr 416
Solid State Bonding in Iron Ore Pellets Apr 411
Sollenberger, C. L., Kirkland, T. G., Bergstrom, B. H., and Mitchell, Will, Jr.: *Analysis of Variables in Rod Milling* Oct 1001
South Africa: corundum industry Apr 403
Oct 1010
Southwest Research Institute: *Lithium—Northeast Brazil Is Potential Source* Sep 897
Spaulding, E. D., Heinrichs, Walter, E., Jr., Thurmond, Robert E.: *Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project* Feb 197
Spodumene production, Brazil Sep 897
Status of Testing Strength of Rocks. Correction July 727
Steel ladders, for driving raises June 618
Stephens, Frank M., Jr. and Langston, Benny: *Collection of Laboratory Dusts* Aug 833
Structure and Mineralization at Silver Bell, Ariz. Nov 1095
Substituted Starches in Amine Flotation of Iron Ore Sep 922
Sulphur-indicator plants in uranium districts Feb 219
Sun, Shiou-Chuan: *Effects of Oxidation of Coals on Their Flotation Properties* Apr 396
Hypothesis for Different Floatabilities of Coals, Carbons, and Hydrocarbon Minerals Jan 67
Sun, S. C., Fisher, H. M., and Snow, R. E.: *Rapid Estimation of Mill Product Purity by Transparency Measurement* Sep 919
Suspension Preheating of Dry Pulverized Materials Apr 407
Swanson, Harry C.: *Mather Mine Uses Pipeline Concrete in Underground Operations* Apr 393
Sweden: drilling practice June 613

T

Taconites: *oxidized* and *nonmagnetic*, definition of terms June 635
see also iron ore
Television, application to conveyor and mining operations July 720
Tennessee: phosphate reserves, source of fluorine Oct 995
Tennessee Copper Co.: *Liquid-Solid Cyclone as a Classifier in the Closed-Circuit Grinding of Concentrates* June 620
Tensile strength of bonded iron ore briquets Apr 412
Testing for Copper and Zinc in Canadian Glacial Soils Jan 57
Texas: hydrocarbon surveys Dec 1206
Thomas, Edward: *Discussion on Use of Wooden Rock Bolts in the Day Mines* Nov 1101
Thurmond, Robert E., Heinrichs, Walter E., Jr., and Spaulding, E. D.: *Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project* Feb 197
Titaniferous concentrates, characteristics of Aug 817
Toron, Luis and Cortes-Obregon, Salvador: *Exploration of the Oaxaca Coal Fields in Southern Mexico* May 505
Trackless Development of an Inclined Limestone Deposit Jan 61
Transvaal: corundum Oct 1011
Trends in Coal Utilization and Their Effect on Coal Marketing Mar 299
Tripp, John G.: *Discussion on Underground Electrocarbonization of Coal and Related Hydrocarbons* Nov 1108
Tumbling Mill Capacity and Power Consumption as Related to Mill Speed July 728
Tungsten carbide tipped drill steel: manufacture of Mar 294
standard lengths used in Sweden June 614
Turnbull, Louis A.: *Discussion on Longwall Mining and Mechanization, with Special Reference to Nova Scotia* Nov 1109

U

Uhley, Robert P. and Scharon, LeRoy: *Gravity Surveys for Residual Barite Deposits in Missouri* Jan 52
Undercutting technique at Jeffrey mine, Canadian Johns-Manville Co. May 513
Underground Electrocarbonization of Coal and Related Hydrocarbons Sep 908
discussion Nov 1108
United Geophysical Co.: *Geophysical Discovery and Development of the Pima Mine, Pima County, Arizona. A Successful Exploration Project* Feb 198
U. S. Atomic Energy Commission: *Origin of Uranium Deposits. A Progress Report* Sep 904
U. S. Bureau of Agriculture: *Phosphate Rock as an Economic Source of Fluorine* Oct 994
U. S. Bureau of Mines: *Federal Coal Mine Safety Act* Jan 54
U. S. Bureau of Mines: safety factor for mine hoisting ropes, definition Oct 989
U. S. Geological Survey: *Botanical Methods of Prospecting for Uranium* Feb 217
U. S. Steel Corp., Michigan Limestone Div.: *Change to Rotary Blasthole Drilling in Limestone Increases Footage, Cuts Time, Saves Manpower* Aug 809
University of British Columbia: *Copper Soil Anomalies in the Boundary District of British Columbia* Jan 49
University of Idaho: *Underground Electrocarbonization of Coal and Related Hydrocarbons* Sep 908
University of Illinois: *Relationship of Geology to Underground Mining Methods* Aug 812
University of Minnesota: *Depth Determinations by Electrical Resistivity* Sep 915
Effect of Variable Surface Layer on Apparent Resistivity Data Dec 1210
Flotation Characteristics of Pyrrhotite with Xanthates Feb 209
Substituted Starches in Amine Flotation of Iron Ore Sep 922
University of Wisconsin: *Adsorption of a Mercaptan on Zinc Minerals* Sep 925
Uranium: botanical methods of prospecting for Feb 217
origin of deposits Sep 904
Use of Wooden Rock Bolts in the Day Mines. Discussion Nov 1101
Utah: phosphate reserves, source of fluorine Oct 995
uranium deposits Sep 904

V

Van Zandt, D. T.: *Change to Rotary Blasthole Drilling Increases Footage, Cuts Time, Saves Manpower* Aug 809
Velasco, J. Ruben and Mulchay, Roland B.: *Sedimentary Rocks at Cananea, Sonora, Mexico, and Tentative Correlation with the Sections at Bisbee and the Swisshelm Mountains, Arizona* June 628
Visman, J.: *Evaluating the Performance of the Cleaning Unit* Oct 1015
Vissac, G. A.: *Discussion on Frontiers in Heat Extraction from the Combustion Gases of Coal* Nov 1112

W

Wada, Masayoshi: *Discussion on Flotation and the Gibbs Adsorption Equation* Nov 1104
Walden, Robert and West, LaMont: *Milling Kentucky Fluorspar Tailings* May 542
Walker, D. R. and Shaw, M. C.: *A Physical Explanation of the Empirical Laws of Comminution* Mar 313
Waller, H. H., Monroe, and Smyth, D. P. R.: *Block Caving at the Jeffrey Mine* May 510
Washington University: *Gravity Surveys for Residual Barite Deposits in Missouri* Jan 52
Wenner Four-Electrode Spread Dec 1210
Wernick, J. H.: *Activation Energies for the Decomposition of Limestone, Dolomitic Limestone, and Dolomite* July 730
Wesner, Adam and Scott, Donald W.: *Properties of Nonmagnetic Taconites Affecting Concentration* June 635
West, LaMont and Walden, Robert R.: *Milling Kentucky Fluorspar Tailings* May 542
Wetting agents in concrete Apr 396
White, W. H. and Allen, T. M.: *Copper Soil Anomalies in the Boundary District of British Columbia* Jan 49
Wilson, G. H.: *Application of Closed-Circuit TV to Conveyor and Mining Operations* July 720
Wilton-Clark, H.: *Productivity in Mining Pitching Seams of the Canadian Rockies* Aug 825
Wyoming: phosphate reserves, source of fluorine Oct 995
uranium deposits Sep 904

X

Xanthates, flotation characteristics of pyrrhotite with Feb 209

Z

Zannaras, J. P.: *Discussion on An Analysis of Mine Opening Failure by Means of Models* Nov 1102
Zinc: Mineral Hill, Ariz. Feb 198
minerals, adsorption of a mercaptan on Sep 925
ores, oxidized, flotation of Apr 416
recovery of cyanide solution values by dust precipitation Dec 1181
testing for in Canadian glacial soils Jan 57